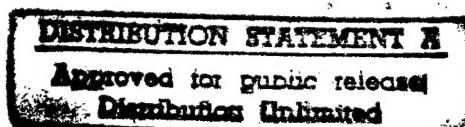


Engineering Report

ALKALINE HYDROLYSIS CONVERSION

of

NITROCELLULOSE FINES



Frank Wolf  
Olin Corporation  
Badger Army Ammunition Plant  
Baraboo, WI 53913

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## EXECUTIVE SUMMARY

The conversion of 1,125,000 pounds of Badger AAP nitrocellulose (NC) fines into a liquid fertilizer is documented in this engineering report. Conversion method was potassium hydroxide (KOH) hydrolysis to digest the nitrocellulose, followed by phosphoric acid pH adjustment. The resultant liquid fertilizer, 1,240,000 gallons, was landspread on Badger AAP fields.

NC fines generated from past production at Badger AAP had laid in storage under water for over 20 years. The "in-process" fines had a nitrogen content of 12.0% to 12.8% nitrogen.

Previous alkaline hydrolysis research was reviewed. Many studies preceded the first production scale, nitrocellulose hydrolysis described by this report. Other potential NC disposal alternatives were identified and reviewed. The only proven, developed technology found was incineration and the prohibited open burning. Now there is a third proven technology - alkaline hydrolysis conversion.

NC removal bids were let with seven companies responding. Most proposals were incineration. Alkaline hydrolysis conversion was the lowest cost alternative.

NC fines were pumped in a water slurry into six 20,000 gallons, agitated reaction tanks and decanted to a final NC content of 11% solids. Approximately 16,800 pounds of NC were processed in each of 68 batches. Excess 50% potassium hydroxide was added to completely digest the nitrocellulose. Two pounds of 50% KOH were added to one pound of NC. KOH addition was controlled to maintain the temperature of the solution at 80°C or less. Digestion was complete with 6 to 10 hours. The reaction tank was allowed to cool and the solution pH adjusted to between 7 and 8 pH with addition of phosphoric acid.

The resultant liquid fertilizer was applied to 1,752 acres of fields, prairies, grasslands and pastures on Badger AAP using conventional liquid fertilizer spreading trucks. Fertilizer plant nutrient value was 1.3% nitrogen (N), 8.0% potassium (as K<sub>2</sub>O) and 0.9% phosphorus (as P<sub>2</sub>O<sub>5</sub>). Total liquid fertilizer volume was 1,240,000 gallons.

Overall application rates were 59 pounds of nitrogen (N) per acre, 350 pounds of potassium (as K<sub>2</sub>O) per acre and 50 pounds of phosphorus (as P<sub>2</sub>O<sub>5</sub>) per acre.

The NC conversion cost \$1,052,500 or 93¢ per pound, was completed with three people, in 110 working days. This NC conversion method met all applicable federal and state, safety and environmental regulations. It was demonstrated alkaline hydrolysis NC conversion, with neutralization and landspreading as a liquid fertilizer, is a proven high volume, economical conversion method.

# I. INTRODUCTION

## A. Background

The Army had over one million pounds of nitrocellulose (NC) pit pulp in storage at Badger Army Ammunition Plant (AAP) generated from past production of nitrocellulose. Nitrocellulose is the major ingredient of propellants produced at Badger AAP. This in-process material is fine nitrocellulose collected in large in-ground concrete settling basins. During production, the fine material would be recycled into the process. When Badger AAP ceased propellant production in 1975, all production facilities were laid away. The "in-process" NC fines were consolidated into two storage basins, accounts 3025 and 9025. The nitrocellulose fines had laid in storage under water for over 20 years.

Nitrocellulose is an important cellulose derivation and has been widely used as a lacquer, film, ink, base, and propellant. NC of high nitrogen content (over 12%) is a main ingredient of propellants, smokeless powder and some explosives. Theoretically fully nitrated NC has a nitrogen content of 14.14%.<sup>1</sup> The chemical structure of fully nitrated NC is shown in Figure 1 below. Theoretical nitration is very difficult to achieve. Most military grade NC is nitrated to a 12.5% to 13.5% nitrogen. Dry NC is extremely flammable. NC is safe and stable when placed in water. The nitrocellulose fines are not considered toxic by the U.S. Environmental Protection Agency.<sup>2</sup>

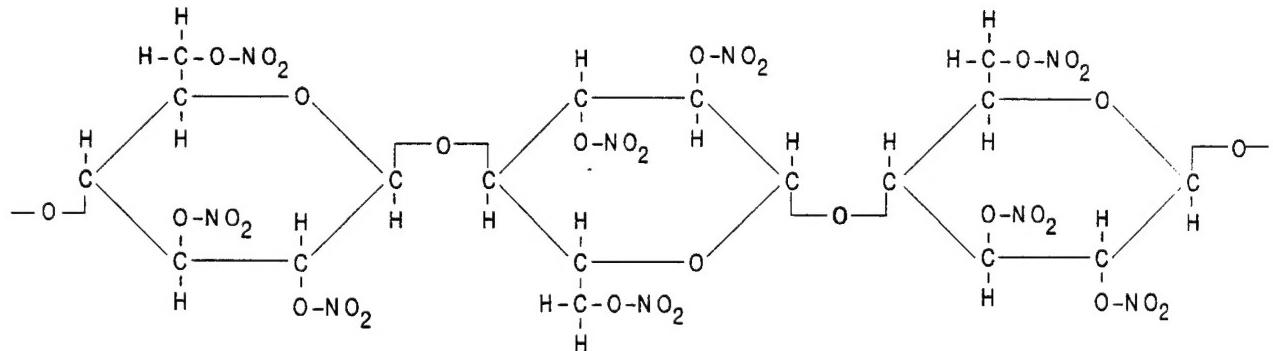


FIGURE 1  
CHEMICAL STRUCTURE OF FULLY NITRATED NITROCELLULOSE

<sup>1</sup> Frank Miles (1955)

<sup>2</sup> EPA (September 1987)

Cellulose is nitrated with a mixture of sulfuric and nitric acid. NC purification follows nitration. The stability of the NC is directly related to the purity. Residual acid causes the impurity. Residuals are removed by boiling, cutting (beating), and poaching (caustic boiling) the nitrocellulose. These purification operations, along with blending and dewatering operations, are accomplished in a water slurry. Large amounts of process water with fine NC are generated. Modernization of the production operations has reduced the amount of water and fines generated, but the quantities are still high. It is reported one pound of NC fines is generated for each 100 pounds of NC produced. Figure 2 is a simplified process flowsheet.<sup>3</sup>

The boiling tub and poacher/blender settling pits are designed to collect NC fines for reuse and recycle within the process. NC fines are of a lower nitrogen content but generally acceptable for use in propellant manufacture. Without production, Badger AAP's NC fines remained within the pits. Badger AAP's NC fines became dirty over the years, with rust, scale, sand and gravel accumulated within the pulp.

Badger AAP's NC fines were still an acceptable NC after 20 years of submerged water storage. Its characteristics are noted in Table 1.

Table 1  
Badger NC Fines Characteristics

|                            |                              |
|----------------------------|------------------------------|
| Cellulose                  | Cotton linters and wood pulp |
| Nitrogen Content           | 12.01% to 12.84% N           |
| Solubility (Ether-Alcohol) | 43% to 100%                  |
| KI Stability               | 65.5°C                       |
| German Stability           | 134.5°C                      |
| Viscosity                  | 3 to 10 sec                  |
| Fineness                   | 52 to 104 mls                |
| Acetone Insoluble          | Trace                        |
| Ash                        | 0.1% to 0.5%                 |

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<sup>3</sup> Badger AAP (12 September 1974)

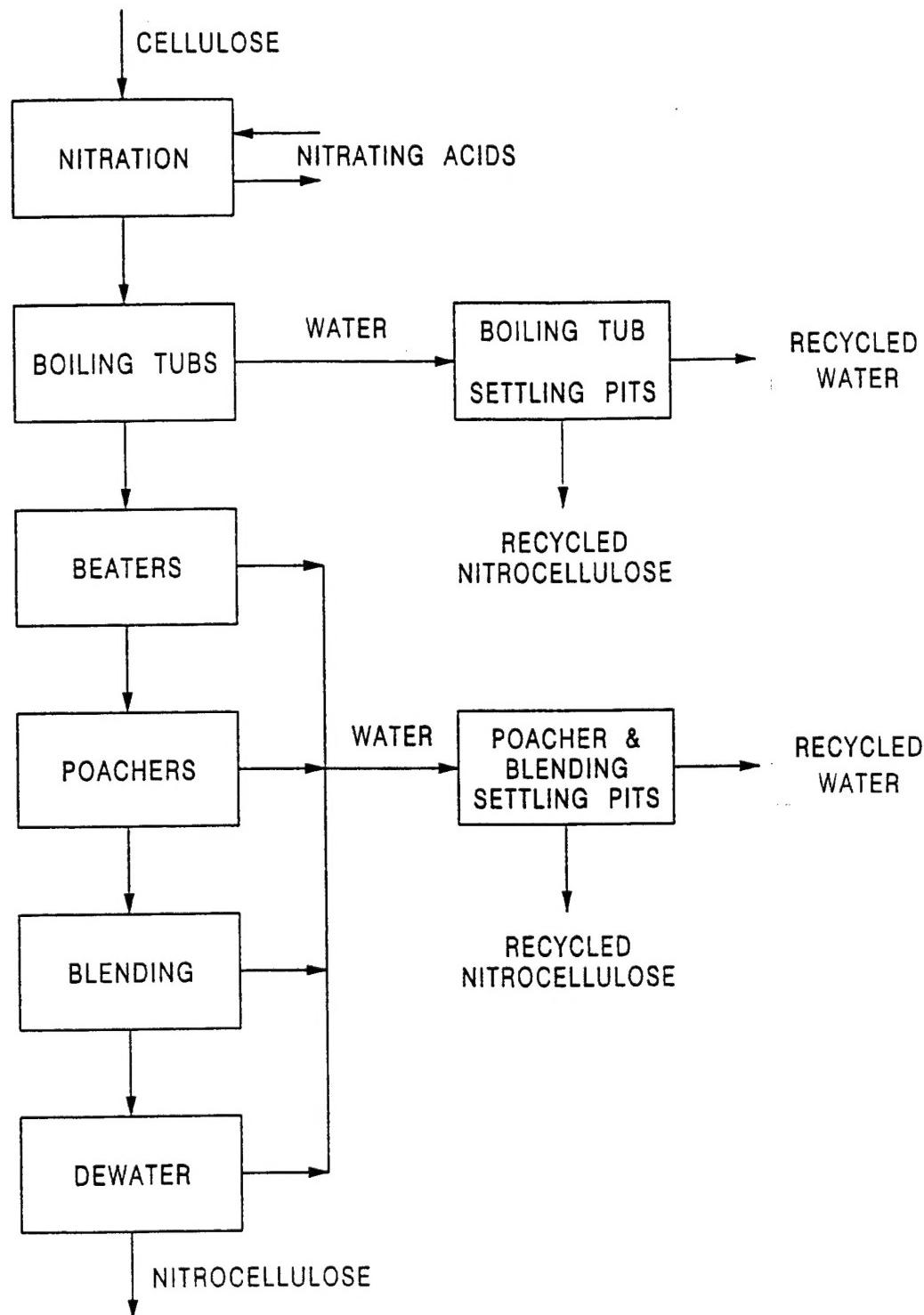


FIGURE 2  
SIMPLIFIED NITROCELLULOSE PROCESS FLOWSHEET

## B. Project Objective

The purpose of this project was to remove all nitrocellulose fines located in two settling basins at Badger AAP, using an environmentally acceptable method.<sup>4</sup> Olin Corporation, Operating Contractor at Badger AAP, was to competitively solicit for the NC removal. The following conditions were required.

1. Subcontractor shall be responsible for all necessary man-hours and materials for removing and disposing of the NC fines and decontaminating the storage basins to a 3X condition, in accordance with Draft IOC Pamphlet 385-5 and Badger AAP Decontamination Administrative Procedure 42-2.
2. Subcontractor will provide the methodology and time phased plan for the removal of NC fines and 3X decontamination.
3. Subcontractor shall prepare any necessary manifests required for shipping material, validating any qualified buyers or certifying actual disposal.
4. Subcontractor is responsible for meeting all applicable federal, state and local safety and environmental regulations.

## C. Previous Alkaline Hydrolysis Work

Previous hydrolysis research was noted in the literature. Many studies preceded the production scale alkaline hydrolysis described in this report. Initial investigation in 1936 by Kenyon and Gray found sizable nitrate levels generated.<sup>5</sup> They used sodium hydroxide at up to a 20% by weight dosage. Increased temperature and alkaline concentration was found to shorten reaction time. Increased nitrocellulose concentration increased reaction time. The reddish-brown color change was also noted. Wolfrom, et al, as quoted in Urbanski (1965), found the products of NC hydrolysis included: inorganic nitrates and nitrites, nitrogen oxides, carbon monoxide (CO), carbon dioxide (CO<sub>2</sub>), ammonia and organic acids.<sup>6</sup>

Wendt and Kaplan in 1976, on bench scale studies with a 3% sodium hydroxide (NaOH) dosage, reported a complete digestion of nitrocellulose at 95°C in ½ hour.<sup>7</sup>

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<sup>4</sup> Contract Modification (December 1996)

<sup>5</sup> W. O. Kenyon and H. Gray (1936)

<sup>6</sup> T. Urbanski (1965)

<sup>7</sup> T. Wendt and A. Kaplan (1976)

Only 1% nitrite was observed in the solution.

Also in 1976 Bissett and Levasseur of the U.S. Army Natick Research and Development Command, hydrolyzed nitrocellulose to give compounds suitable as fertilizer.<sup>8</sup> It was found that a 5% suspension of NC was completely decomposed when heated in a 10% aqueous ammonia solution for 8 hours at 75°C. Hydrazide was also studied as a potential digestion agent. At the same time, Blum, also from the U.S. Army Natick Research and Development Command, was investigating sodium hydroxide nitrocellulose hydrolysis.<sup>9</sup> He found through infrared analysis, the hydrolysis products to be a mixture of many compounds, the bulk of which are nitrated hydroxy acids.

Balasco, et al, in 1987 presented alkaline hydrolysis as the most flexible and cost effective technology for disposal of NC fines.<sup>10</sup> A proposed process concept was given with little specific data. Capital and operating costs were presented.

Hirayamo and Smith reported at USATHAMA's 13th Annual Symposium in 1988 of their pilot scale hydrolysis demonstration using a 4% NaOH solution.<sup>11</sup> NC concentration was 2% by weight with all research carried out under ambient conditions. Their resultant solution contained soluble nitrate and nitrite compositions of 360 and 520 mg/l, respectively.

Other hydrolysis investigations were reported in November 1993 at the U.S. Army - Purdue University, "Nitrocellulose Fines Separation and Treatment Workshop". Alleman, Quivey and Equelva of Purdue University and Kim of the U.S. Army Construction Engineering Research Laboratory cooperated in extensive hydrolysis research.<sup>12</sup> They concluded that low caustic dosage could achieve nearly complete digestion in a short reaction time with most of the carbon solubilized and significant levels of nitrite and nitrate released. The work involved sodium, potassium and calcium hydroxide treatments up to 10% dosages.

The Center for Environmental Engineering, Stevens Institute of Technology, Hoboken,

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<sup>8</sup> F. Bissett and L. Levasseur (September 1976)

<sup>9</sup> Aaron Blum (September 1976)

<sup>10</sup> A. Balasco (September 1987)

<sup>11</sup> L. Hirayama and L. Smith (1988)

<sup>12</sup> J. Alleman et al (1993)

NJ presented another hydrolysis paper at the 1993 U.S. Army - Purdue Workshop.<sup>13</sup> The researchers digested a nitrocellulose propellant with different sodium hydroxide solutions at various temperatures and reaction times. Reaction time, temperature, and hydroxide concentration were found to be functions of the digestion rate. Increased variables increased the digestion rate. The work was updated in a paper given at the 1994 Aerospace Environmental Technology Conference in Huntsville, AL.<sup>14</sup> Data suggested nitrocellulose hydrolysis is a single step process where nitrates and nitrites are formed directly from the cleavage of the parent molecule.

Detailed alkaline hydrolysis research was performed by David Quivey and reported in his doctoral thesis of December 1994<sup>15</sup>. His hydrolysis studies were designed to evaluate the effects of caustic type, caustic dosage, temperature and solids concentration. It was found KOH was a slightly more effective agent than NaOH and both were much more effective than the other hydrolysis agent studied Ca(OH)<sub>2</sub>. Increased caustic dosage and increased hydrolysis temperature was found to significantly increase digestion rate. Caustic dosage of 10% NaOH did not increase digestion rate appreciably over the 5% dosage. Dilute solids concentrations produced the more effective digestion rates. Initial solids concentrations studied were 1, 3, 5 and 7% nitrocellulose. Solids concentrations did not have as much effect on digestion rate as dosage or temperature. The alkaline hydrolysis process was shown to completely solubilize a 10% NC slurry via a predictable reaction following pseudo-first-order kinetics. Optimum hydrolysis conditions for solubilization based on non-linear optimization of the theoretical solubilization equation, were found to be a 60°C. temperature, 20% caustic concentration and a 12 hour reaction period.

Other recent research on alkaline hydrolysis to destroy energetic materials including nitrocellulose was reported in a paper given by T. M. Benziger et al, of Los Alamos National Laboratory at the 1993 Incinerator conference.<sup>16</sup> A second paper on follow-up hydrolysis work at Los Alamos was given by a colleague Terry Spontarelli, et al, at the 1994 ADPA Energetic Materials Technical Symposium.<sup>17</sup> These papers describe a safe, simple and inexpensive method to convert energetic materials (RDX, HMX, TNT, NC, NG and NQ) into non-energetic materials. Materials were hydrolyzed with aqueous sodium hydroxide or ammonia. Material was decomposed at 60° to 150°C.

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<sup>13</sup> C. Christodoulatos et al (1993)

<sup>14</sup> M. Sidhoum, et al (11 August 1994)

<sup>15</sup> D. Quivey (December 1994)

<sup>16</sup> T. M. Benziger, et al (1993)

<sup>17</sup> T. Spontarelli, et al (24 March 1994)

after 4 to 5 hours. Decomposition products included organic and inorganic salts with mostly nitrous oxide gaseous emission. These products will require further treatment.

Recent laboratory work by Ron Borcherding and Dean Wolbach was completed in 1995.<sup>18</sup> Hydrolysis of nitroglycerin, nitramine (HMX) and ammonium perchlorate was studied. The propellants (100 grams) were reacted with two molar NaOH. Detailed results were not reported. A preliminary design, with process flow diagram, for a 200 pound per 8-hour shift process was developed.

The latest reported research found was by Arctech, Inc. of Chantilly, VA, who has developed a proprietary process of recycling propellants and explosives into fertilizers.<sup>19</sup> The one step process involves hydrolysis with solutions containing ACTOSOL® humic acids and the subsequent conversion of the hydrolysis products to fertilizers. Arctech, Inc. was awarded U.S. Patent 5,538,530 on July 23, 1996, covering this novel process invention.

A summary of previous caustic hydrolysis research is shown in Table 2 and compared with actual operations observed in this reported production operation. Actual operation's alkaline dosage was stronger than most research dosages. Reaction temperature, solid concentration and reaction time of actual operations were within the range of research parameters.

#### D. Potential Disposal Methods

A review of disposal technologies has identified many candidate alternatives whose time of appearance in commercial scale varies from currently available to more than five years in the future. Alkaline hydrolysis technology was discussed in a previous section and not further discussed here. This review concludes specific thermal treatment is the only technology that could efficiently treat a large quantity of material. A summary of potential alternatives is presented in Table 3. Status of alternative development is noted in that table. Alternatives found in the literature search are summarized further in the following paragraphs. Several of these citations are alternative summaries and assessments.

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<sup>18</sup> Ron Borcherding and Dean Wolbach (14 July 1995)

<sup>19</sup> U.S. Patent Number 5,538,530 (23 July 1996)

**Table 2**  
**Historical Research**

| Author                | Year | Hydroxide            | Dosage     | Temp °C    | Reaction Time-Hrs | Solid Concentration |
|-----------------------|------|----------------------|------------|------------|-------------------|---------------------|
| Kenyon & Gray         | 1936 | Sodium               | 1 to 20%   | 30, 60     | ½ to 1100         | --                  |
| Wolfrom et.al.        | 1965 | --                   | --         | --         | --                | --                  |
| Wendt & Kaplan        | 1976 | Sodium               | 3%         | 95         | ½                 | 5%                  |
| Blum                  | 1976 | Sodium               | 3%         | 90 to 95   | ½                 | 5%                  |
| Bissett & Levasseur   | 1976 | Ammonia              | 5 to 30%   | 25 to 98   | 1 to 120          | 5, 10%              |
| Balasco et.al.        | 1987 | Sodium               | 5%         | 65 to 95   | 3                 | 10 to 25%           |
| Hirayama & Smith      | 1988 | Sodium               | 4%         | 25         | Hrs               | 2                   |
| Alleman et.al.        | 1993 | Na, K, Ca            | 1 to 10%   | 25, 35, 50 | 1 to 24           | 1 to 7%             |
| Christodoulatos et al | 1993 | Sodium               | 5, 10, 15% | 35 to 95   | ½ , 1             | --                  |
| Benziger              | 1993 | Na & NH <sub>4</sub> | 8%         | 60 to 150  | 4-5               | 3 to 20%            |
| Spontarelli           | 1993 | Na & NH <sub>4</sub> | 8%         | 60 to 150  | 4-5               | 3, 10%              |
| Quivey                | 1994 | Na, K, Ca            | 1, 5, 10%  | 25 to 70   | 1 to 24           | 1 to 7%             |
| Sidhoum, et al        | 1994 | Sodium               | 5 to 15%   | 35 to 95   | 1                 | 1%                  |
| Borcherding           | 1995 | Sodium               | --         | --         | --                | 5%                  |
| Arctech               | 1996 | Potassium            | 20%        | < 40       | 50 to 100         | 5%                  |
| This project          | 1997 | Potassium            | 5 to 25%   | < 80       | 6 to 10           | 4 to 16%            |

**TABLE 3**  
**HAZARDOUS WASTE DISPOSAL ALTERNATIVES**

| Alternative                        | Status   |
|------------------------------------|--|
| <b>Thermal Treatment</b>           |  |
| Open Burning/Open Detonation       | Not allowed                                    |
| Incineration                       | Well developed process                         |
| Wet Air Oxidation                  | Lab scale tests                                |
| Low Temp. Thermal Description      | Pilot scale tests not successful               |
| Induction Coupled Plasma           | Lab scale tests                                |
| <b>Biological Treatment</b>        |  |
| Aqueous - Phase Bioreactor         | Lab scale tests were mixed                     |
| Composting                         | Pilot scale tests were successful, feasible    |
| Land Forming                       | Pilot studies not successful                   |
| White Rot Fungus                   | Pilot studies were mixed                       |
| Direct Fertilizer                  | Lab scale tests                                |
| Feed Supplement                    | Lab scale tests                                |
| <b>Physical/Chemical Treatment</b> |  |
| Ultraviolet Oxidation              | Only low concentrations                        |
| Activated Carbon                   | Only low concentrations                        |
| Reuse/Recycle Options              | Not always possible or developed, but feasible |
| Solvent Extraction                 | Well developed, but costly                     |
| Supercritical Water Oxidation      | Lab scale, expensive                           |
| Fuel Supplement                    | Small quantities                               |
| Cryogenic                          | Lab scale tests                                |
| Adams Sulfur Process               | Lab scale tests                                |
| Dimethylsulfoxide                  | Lab scale tests                                |
| Base Hydrolysis                    | Lab scale tests                                |
| Molten Salt                        | Lab scale tests                                |
| Electrochemical Oxidation          | Lab scale tests                                |
| Hydrothermal                       | Lab scale tests                                |

An early study of treatment alternatives was completed by J. M. Genser, et al, in 1977.<sup>20</sup> Twenty-four hazardous waste streams were studied of which three were explosive wastes. Rotary kilns were selected for nineteen streams including explosive streams. Extensive cost estimates and economic analysis were presented. Explosive disposal costs ranged from 12¢ to 70¢ per pound.

The U.S. Army Corps of Engineers Toxic and Hazardous materials Agency published the third edition of their handbook "Installation Restoration and Hazardous Waste Control Technologies" in 1992.<sup>21</sup> The purpose of the handbook is to provide a reference of pertinent and current treatment technologies. Handbook information was derived from personal interviews with personnel directly involved in search, development and implementation of new and effective methods to accomplish the following: restoration of contaminated soils, groundwater and structures, and the minimization of the generation of hazardous waste materials. One hundred fifty-seven technical notes were summarized with fifty-one pertaining to hazardous waste control. Most of the notes referred to minimization, recovery and reuse.

Steven Torma, et al, presented a paper at the Annual Meeting of the Minerals, Metals & Materials Society, reviewing some of the technologies available for recycling energetic projectiles.<sup>22</sup> Recycling technology involves dismantling and separation of ammunition components into recyclable metals, plastics, paper and explosive materials. Explosives can be recycled to be used for industrial purposes in the mining industry. Propellants may be used in the agricultural industry as fertilizer or incinerated to produce heating steam. Other technologies discussed were supercritical water oxidation, plasma arc centrifugal furnace and cryofracture. Munitions demilitarization in 1994 was still mostly open burning/open detonation (82%) with incineration as the next most utilized method at only 10%. Other methods are used in less than 5% of the disposals.

The American Defense Preparedness Association sponsored an international symposium on "Energetic Materials Technology" March 21-14, 1994 at the Clarion Plaza Hotel, Orlando, FL. Many papers were given on demilitarization technology focussing on reuse/recycle methods. The next five literature citations are papers given at the symposium.

Larry Sotsky, Project Leader with the Explosives and Demilitarization Section, U.S.

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<sup>20</sup> J. M. Genser, et al (2 September 1977)

<sup>21</sup> Michael Madden and William Johnson (November 1992)

<sup>22</sup> Steve Torma, et al (3 March 1994)

Army Armament Research, Development and Engineering Center presented a paper describing three tasks developed to treat "difficult" energetic materials.<sup>23</sup> A state-of-the-art plasma arc furnace was used to destroy pyrotechnic munitions at a DOE test site in Butte, Montana. Pyrotechnic compositions have also been destroyed with DRE > 99.99 + % using supercritical water oxidation technology. The third task evaluated the recycle/reuse of red phosphorus/butyl rubber smoke grenade material. Initial results are favorable.

R. Eric Dotseth and David W. Ling of Mason & Hanger - Silas Mason Company, Inc. Presented a paper to describe expanding the demilitarization and disassembly capabilities of the Iowa Army Ammunition Plant in support of the U.S. Army's efforts to move from open burning/open detonation toward resource recovery.<sup>24</sup> This effort to expand the demilitarization capability included disassembly, energetic material removal, confined detonation, controlled incineration, and overall waste and hazard classification. Several processes and methods have been developed to perform this safely and environmentally. Their paper described the methods of disassembly and energetic material removal for high explosive and anti-personnel cartridges, 90mm through 106mm. These cartridges represent a significant portion of the munitions inventory slated for demilitarization, with a wide variety of explosive and propellant loads. A description of the decision process for determining the process for the demilitarization line, and the actual end result was discussed. Additionally, actual operating experience was described to show what went as planned and what obstacles arose during extended operation.

Thomas J. Schilling, et al, presented a paper that reviewed the reprocessing and reuse programs at Crane Naval Surface Warfare Center in the development of commercial uses for surplus explosives, propellants and their constituents.<sup>25</sup> Emphasis was on RDX and HMX reuse in oil field services.

A high performance blasting agent for metal bonding applications was developed. High valued HMX was extracted for perforating charge applications. Recoverable yields were > 98% with a purity > 99.5%. A surplus energetics reprocessing pilot plant was being designed to manufacture 125 tons annually of blasting agent from surplus explosives.

A paper was presented to describe how the Naval Surface Warfare Center, has concentrated on reclaiming the valuable energetics with subsequent reformulating for

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<sup>23</sup> Larry Sotsky (24 March 1994)

<sup>24</sup> R. Eric Dotseth and David W. Ling (24 March 1994)

<sup>25</sup> Thomas J. Schilling, et al (24 March 1994)

commercial applications.<sup>26</sup> Technology Development Inc. (TDI), Rolla, MO and TPL, Inc., Albuquerque, NM have demonstrated the feasibility of using reclaimed military explosives as efforts have concentrated on reformulation to produce a commercial mining explosive, while TPL has concentrated on producing a metal brazing explosive. In both cases, various reclaimed PBXs and other explosives have been reformulated and tested to produce explosives of equal or superior performance to current commercial explosives. Work has also been initiated to recover RDX and HMX from military explosives and propellants and to qualify the RDX and HMX for commercial applications. Tests conducted by TPL, Inc. Indicate that a feed supplement for ruminant animals and a slow nitrogen release fertilizer can be generated from surplus Navy gun propellants. The feasibility of using surplus gun propellants in a novel oil and gas well stimulation process was also demonstrated.

Another paper cited presented at the "Energetic Materials Technology" symposium was given by D. S. Wulfman of D. S. Wulfman and Associates, Inc.<sup>27</sup> His paper discussed the results of ongoing reformulation studies begun in the late 1980s. Field applications of polymer bonded explosives were described. Reformulation can be accomplished with minimal environmental impact and the resulting explosives are in many instances theoretically "better" explosives than the original PBXs.

William Melvin and Roger Shippey discussed the latest Army missile demilitarization program with a priority on recycle and reuse at JANNAF's Joint Meeting in March 1997. They discussed how the latest technologies are integrated into their Redstone Arsenal Recycling System.<sup>28</sup> Dan Burch, et al, discussed the Navy's latest technologies to maximize resource recovery and recycle (R3) of energetic materials.<sup>29</sup> The basic research has been completed and most technologies are now in the demonstration phase. Potential reuse include commercial mining explosives, metal brazing, explosives, gun propellant components, fertilizer, oil/gas simulations and sporting powder.

Research as part of a Small Business Innovative Research sponsored by the naval Sea Systems Commands Demilitarization program found two agricultural applications for reuse of NC based propellants.<sup>30</sup> These laboratory investigations were performed by

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<sup>26</sup> Dan Birch and Mike Johnson (24 March 1994)

<sup>27</sup> D. S. Wulfman, et al (24 March 1994)

<sup>28</sup> William Melvin and Roger Shippey (March 1997)

<sup>29</sup> Dan Burch, et al, (March 1997)

<sup>30</sup> H. Stoller (November 1993)

TPL, Inc. Greenhouse/Multi-Pot studies with sorghum found nitrocellulose can release nitrogen to improve plant growth. Nitrocellulose was loaded directly to the potted plant at loadings of 35 to 280 pounds per acre. The second TPL, Inc. research investigated NC as a ruminant animal (cows, sheep, goats, etc.) nitrogen feed supplement to aid the animals rumen microbe digestion. Feed supplement application was believed to be demonstrated, but not unambiguously.

A Canadian Armed Forces sponsored paper presented a study on biodegradation of energetic compounds (RDX, TNT, NC and GAP).<sup>31</sup> Successful degradation occurred in concentrations up to 27,000 mg/kg. RDX mineralization rate reached 5 mg/kg/day when utilized as a nitrogen source under aerobic conditions.

The U.S. Army Environmental Center (USAEC) has sponsored much research on the composting biodegradation of energetic materials. Composting is a process in which materials are biodegraded by micro-organisms, usually at an elevated temperature. Research conducted since 1986 by USAEC has shown that variety of nitroaromatic explosives in soils can be treated by composting.<sup>32 33 34 35 36 37</sup> Additional research has shown the treatment of NC in soils is technically feasible.<sup>38 39</sup> A recent economic analysis had shown that composting of NC fines is an economically feasible treatment alternative.<sup>40</sup> Finally, a recent study of the regulatory and logistical feasibility of NC fines composting was completed.<sup>41</sup> All the research indicates NC composting is a potential NC disposal or recycle method.

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<sup>31</sup> S. Thiboutot (24 March 1994)

<sup>32</sup> Altantic Research Corporation (1986)

<sup>33</sup> R. Williams, et al (March 1989)

<sup>34</sup> R. Williams, et al (September 1989)

<sup>35</sup> R. Williams, et al (November 1991)

<sup>36</sup> C. Breed (March 1991)

<sup>37</sup> W. Lowe, et al (August 1993)

<sup>38</sup> Atlantic Research Corporation (1986)

<sup>39</sup> R. Williams, et al (March 1989)

<sup>40</sup> Roy Weston, Inc. (1995)

<sup>41</sup> W. Lowe (December 1995)

A Sandia National laboratory report by Costanzo A. La Jeunesse, et al, describes the concept of a supercritical water oxidation reactor to destroy colored smoke, spotting dye and pyrotechnic munitions.<sup>42</sup> Process and equipment operation parameters, process flow equations or mass balances and utility requirement for wastes are developed in this report. Two conceptual designs are developed with all process and instrumentation detailed. Concept is based on bench scale reactor work. Capital cost for a 20 lb/hr plant is \$789,500 (1993\$). Another Sandia National Laboratory poster presentation at the 1994 Joint USA-Russia Energetic Material Technology Symposium in Livermore, CA on May 18-25, 1994 further described the cryoclyning demilitarization process.<sup>43</sup>

Further supercritical processing work using supercritical carbon dioxide was described by Robert Farncomb and George Nauflett at the "Environmentally Sound Processing Technology Workshop" sponsored by Joint Army-Navy-NASA-Air Force (JANNAF) in July 1995.<sup>44</sup> Successful data from a 2 liter extraction vessel was discussed.

The latest supercritical carbon dioxide processing developments were reported by James Coccharo in March 1997 at a JANNAF Joint meeting. The supercritical process has developed into pilot scale research.<sup>45</sup>

SRI International has conducted a study on the destruction of energetic materials in hydrothermal media near the critical temperature of water.<sup>46</sup> The target materials included AP, RDX, HMX, NG, TNT, and CL-20. The bench scale work was conducted with liquid water at autogenous pressures at temperatures over the range 70° to 350°C. It was found the simple reaction with water should provide a process yielding 5-nines destruction at or below 350°C. with residence times of 100 - 200 seconds. Preliminary cost estimates for a 300 lb/hr plant were \$700,000 with an estimated operating cost of \$700/ton.

G. Ostrom and R. Hollins of the China Lab Naval Air Warfare Center have investigated the possible catalytic effect of simple salts on the destruction of energetic material using hydrothermal degradation.<sup>47</sup> Test results indicate complete destruction within minutes, with no detectable by-product species.

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<sup>42</sup> C. A. La Jeunesse, et al (November 1993)

<sup>43</sup> L. L. Whinnery, et al (25 May 1994)

<sup>44</sup> Robert Farncomb and George Nauflett (July 1995)

<sup>45</sup> James Coccharo, et al (March 1997)

<sup>46</sup> David S. Ross (24 March 1994)

<sup>47</sup> G. Ostrom and R. Hollins (July 1995)

Burns and Roe, Defense and Aerospace Division, have developed the Adams Process, a potential chemical method that reacts organic materials in an atmosphere of elemental sulfur vapor (typically 450° to 600°C.<sup>48</sup> In this process, the organic materials are rapidly reacted to form a variety of simple sulfur compounds. The gaseous products are readily recovered or treated in conventional off-gas cleanup. Gaseous emissions from cleanup can be recycled back to the reactor. Bench scale tests on explosives has been performed with destruction complete within a 4-hour time frame. DRES's could be as high as 99.9999%.

HMX reclaimed by a dimethyl sulfoxide (DMSO) solvent recovery method was discussed in a paper presented by Randall W. Hurd of Mason & Hanger - Silas Mason C, Inc.<sup>49</sup> Laboratory work produced HMX product of 99.3% purity.

Millard M. Garrison of Alliant Techsystems, Inc. and John Serino of Plasma Technology Inc. presented a paper that described a thermal destruction treatment with an argon induction coupled plasma torch at 10,000°C.<sup>50</sup> DRE is up to 99.9999%. No additional waste streams are generated. Initial test work was done at Drexel University. Several other groups of researchers are working on hot plasma techniques.<sup>51</sup> The MIT Plasma Center in Cambridge, MA houses two 30 KW plasma arc furnaces where hazardous material moves through a 10,000°C. plasma arc developed by graphite electrodes. Researchers at Georgia Institute of Technology are working on a plasma torch to be used for in-situ disposal methods. It is felt these plasma techniques can be cost competitive.

Three documents were found describing waste energetic materials used as a fuel supplement. The initial citation is the 1988 report of W. M. Bradshaw of Oak Ridge National Laboratory.<sup>52</sup> His bench scale work presented proof-of-principle tests in a 300 KW combustion furnace firing up to 40% TNT or 37% Comp B in toluene/fuel oil mixtures. The second citation is an article in the Journal of Hazardous Materials by Craig A. Myler, et al.<sup>53</sup> Their laboratory and bench scale work further verify the principle while economics presented show a positive advantage. The last fuel supplement citation also by Craig A. Myler presents additional results of their test

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<sup>48</sup> James R. Hendrichs and Joseph S. Klimek (24 March 1994)

<sup>49</sup> Randall W. Hurd and George L. Clink (24 March 1994)

<sup>50</sup> Millard Garrison and John Serino (24 March 1994)

<sup>51</sup> William D. Siuri (October 1994)

<sup>52</sup> W. M. Bradshaw (August 1988)

<sup>53</sup> Craig A. Myler, et al (1991)

work.<sup>54</sup> Their most recent work will utilize a 498 KW boiler.

Based on technology originally developed at Lawrence Livermore National Laboratory, E. O. Systems, Inc. of Palo Alto, CA has developed a promising technique referred to as mediated electrochemical oxidation or MEO.<sup>55</sup> MEO pumps liquid wastes through a closed loop system and destroys it in an acid electrolyte such as sulfuric acid. The waste materials are broken down into water and carbon dioxide. Upon completion, the acid can be regenerated and reused.

The last hazardous waste disposal alternative citations found were about the destruction of materials using molten salt. Both citations on the molten salt were by Ravindra S. Upadbye, et al of Lawrence Livermore National Laboratory bench scale work.<sup>56 57</sup> The molten salts are typically mixtures of alkali or alkaline earth carbonates and halides. The salts provide excellent heat transfer and reaction media, catalyze oxidation of organics and neutralize acid gases by forming stable salts such as sodium chloride. They have successfully and safely destroyed slurries of 35% HMX, RDX, PETN and TATB in mineral oil and 50% of the above in water. The temperature of the molten salt is varied between 400° to 900°C. They have also destroyed XM-46 liquid propellant. Destruction rates were 500 to 1000 grams per hour.

This section on review of waste disposal technologies concludes with an article by Timothy J. Tope and Walker F. Howell of the Radian Corporation, Oak Ridge, TN.<sup>58</sup> Their article presents the role of open burning/open detonation, and also discusses the disposal technologies currently applied and being developed for demilitarization purposes, and an analysis of advantages and limitations of these technologies. A comparison of their treatment alternatives is presented in Table 4. This comparison was conducted using five key criteria: treatment effectiveness and application, environmental impacts/regulatory concerns, safety concerns, costs and degree of development. Table 4 indicates that most alternatives are not capable of treating explosive waste on a large scale bases.

The only developed technology other than open burning/open detonation was incineration. Now there is a third proven technology, caustic hydrolysis, as shown in this report.

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<sup>54</sup> Craig A. Myler, et al (1994)

<sup>55</sup> William D. Siuri (October 1994)

<sup>56</sup> Ravindra S. Upadbye and Bruce E. Watkins (March 1994)

<sup>57</sup> Ravindra S. Upadbye, et al (25 April 1994)

<sup>58</sup> Timothy J. Tope and Walker F. Howell (Summer 1994)

**Table 4**  
**Comparative Analysis Between Treatment Technologies**

| Treatment Technology | Treatment Effectiveness & Application   | Environmental Impacts Regulatory Concerns  | Safety Concerns  | Costs  | Degree of Development   |
|----------------------|---|--|--|--|---|
| Recovery/Recycling   | Limited, reclamation represents only an intermediate step in overall recovery process                 | Dependent upon process; potentially more threatening; RCRA implications throughout process.                        | Poses a greater risk to workers performing operations.   | Requires independent cost analysis but typically presents greater costs than OB/OD because processes tends to be labor and energy intensive. | R&D programs in early stages.   |
| Incineration         | Limited despite variations for waste munitions; incineration has associated maintenance difficulties. | Destruction efficiency comparable to OB/OD; extensive technical permitting.  | Poses potentially less risk problem because destruction process is more controlled.  | Significantly higher operating and capital expenses.   | Developed technology but treatability studies required for each waste stream. |
| Popping Furnace      | More limited than OB/OD since feed rate controlled and designed for small arms munitions.             | Destruction efficiency makes this option comparable to OB/OD for small arms; has some special regulatory concerns. | Poses potentially less risk problem since destruction process is more controlled and is usually limited to smaller munition items. | Higher capital and operating expenses.   | Further development required for feed systems.                                |
| Reduction/Conversion | Options have shown limited application or success.  | Impacts from these alternatives not documented; expected to be slightly less of an environmental hazard.           | Controlled processes pose less risk.   | Limited data make cost comparison difficult.   | R&D programs in early stages.   |
| Cryofracture         | Limited since process is only an intermediate step in overall treatment                               | Initial data indicates less of an environmental hazard; regulatory requirements unknown.                           | Under development; safety factors cannot be evaluated at this time.  | Insufficient data to estimate operating costs; capital costs most likely higher.   | R&D programs in early stages.   |
| Biodegradation       | Testing has included only selected reactive wastes.   | Biodegradation is generally considered a low impact alternative.   | Does not involve thermal destruction; would therefore be expected to be safer.   | Limited data makes cost comparison difficult.  | R&D programs in early stages.   |

## I I. MATERIALS, EQUIPMENT AND METHODS

Olin Corporation solicited proposals from 18 firms to remove the NC fines. It was indicated both positive and negative bids would be considered. Responses were received from seven companies. Most vendors proposed drumming the NC fines, then shipment to an incinerator for final disposal. Bids ranged from \$2 to \$6 million for this disposal method. Another vendor proposed direct NC landspreading as a water slurry and immediately plowed under. Corn would then be planted on the freshly plowed land. A composting bid was not received.

The low cost proposal was selected as the NC fines conversion method. Potassium hydroxide hydrolysis treatment followed by landspreading of the resultant liquid fertilizer was the selected method. Bioremediation Service, Inc. (BSI), Portland, OR, was awarded a contract for \$750,000 for the conversion of the one million pounds of bone-dry nitrocellulose. An additional charge of \$700 per each 1000 pounds of NC over the initial million pounds was negotiated.

### A. Materials

#### 1. Nitrocellulose Fines

The nitrocellulose fines were described in paragraph 1A and Table 1 as characteristic of military grade nitrocellulose of about 12.5% nitrogen content, but very dirty. NC storage was under water at an average bulk density of 12.7 pounds of bone-dry nitrocellulose per cubic foot. Total NC slurry volume was 650,000 gallons. The total volume of water cover and slurry in the two basins was 1.2 million gallons. 1,125,000 pounds of bone-dry nitrocellulose were processed.

#### 2. Potassium Hydroxide (KOH)

Potassium hydroxide in a 50% water solution was used to hydrolyze the NC fines. This commercial grade KOH was delivered in 45,000 to 50,000 pound loads via truck trailer. A total of 2,132,000 pounds KOH solution was consumed.

#### 3. Phosphoric Acid ( $H_3PO_4$ )

Phosphoric acid at 75% concentration was used to neutralize the caustic hydrolyzed solution. This commercial grade acid was also delivered in truck load quantities with a total of 170,000 pounds of solution being consumed.

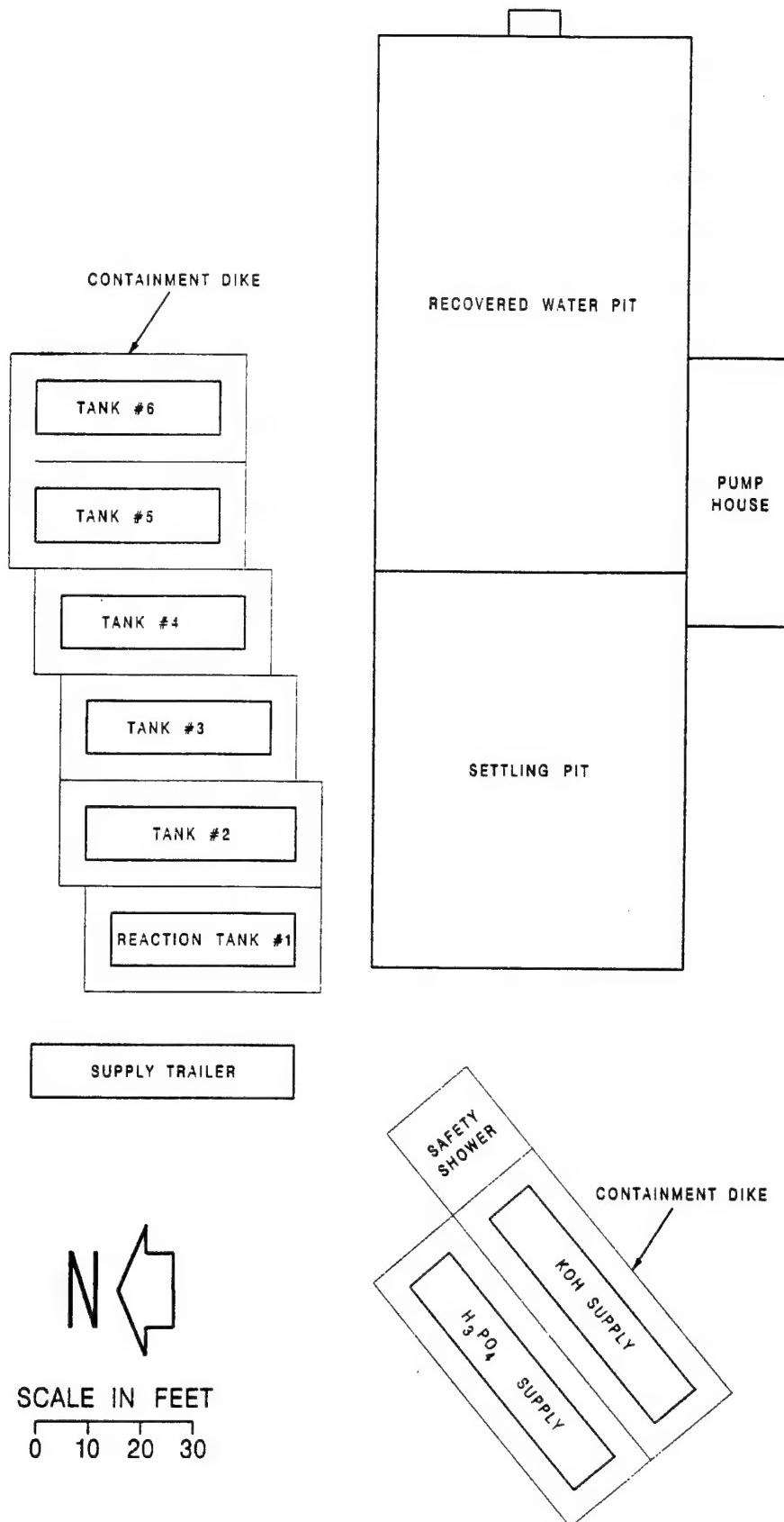


FIGURE 3 PROJECT SITE PLAN

## B. Equipment

Process equipment used to hydrolyze the NC fines was located around storage basin, account 3025, as shown in Figure 3.

### 1. Reaction Tanks

Reaction tanks were of two types. There were five 20,000 gallon enclosed "Baker Tanks" constructed of  $\frac{1}{2}$ " thick mild steel. Dimensions were 35 feet long, 7 feet-10 inches wide and 10 feet tall. Bottom shape was irregular and four 10 HP agitators were positioned as shown on Figure 4. One "Rain for Rent" tank was also used of a slightly larger configuration (21,000 gallon) as shown on Figure 4. Its dimensions were 40 feet long, 7 feet-10 inches wide and only 8 feet tall. Three 10 HP agitators slurried its contents. Access hatches and nozzles were located as noted on Figure 4. Agitators were a solid 1 inch diameter shaft with four 3 inch flat blades 2 feet long at two locations on the shaft - bottom and the midpoint.

### 2. Pumps

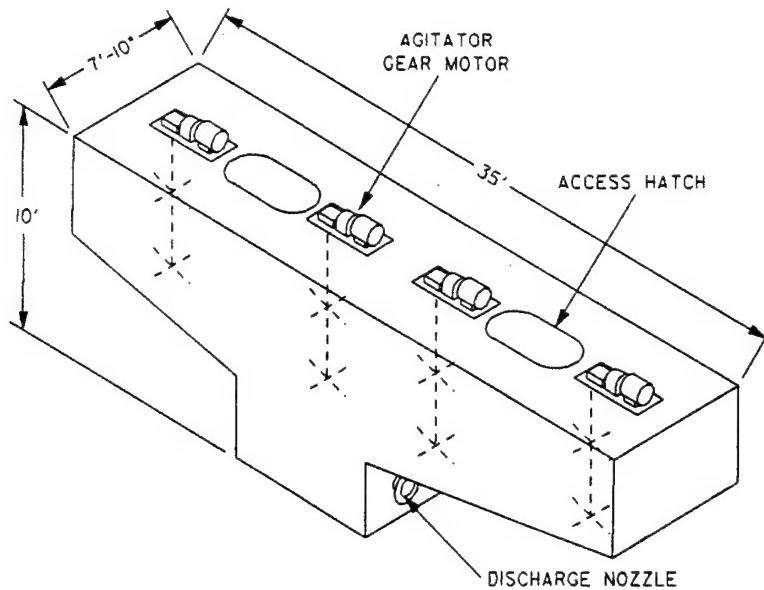
An agricultural manure pump driven by a farm tractor power takeoff (PTO) was used to circulate the NC fines within the storage basins, to pump the NC fine slurry into the reaction tanks, and to pump the liquid fertilizer out of the reaction tank into the fertilizer spreading trucks. Two identical manure pumps were used. These pumps are normally used to pump out liquid farm manure storage lagoons. The direct driven pump impeller was 30" in diameter and 4" wide. Operating pump capacity was 100 to 500 gallons per minute depending on PTO speed. Smaller electric and gasoline pumps were used to decant or pump process water from the reaction tanks to the storage basins or within and out of the basins.

### 3. Vacuum Truck

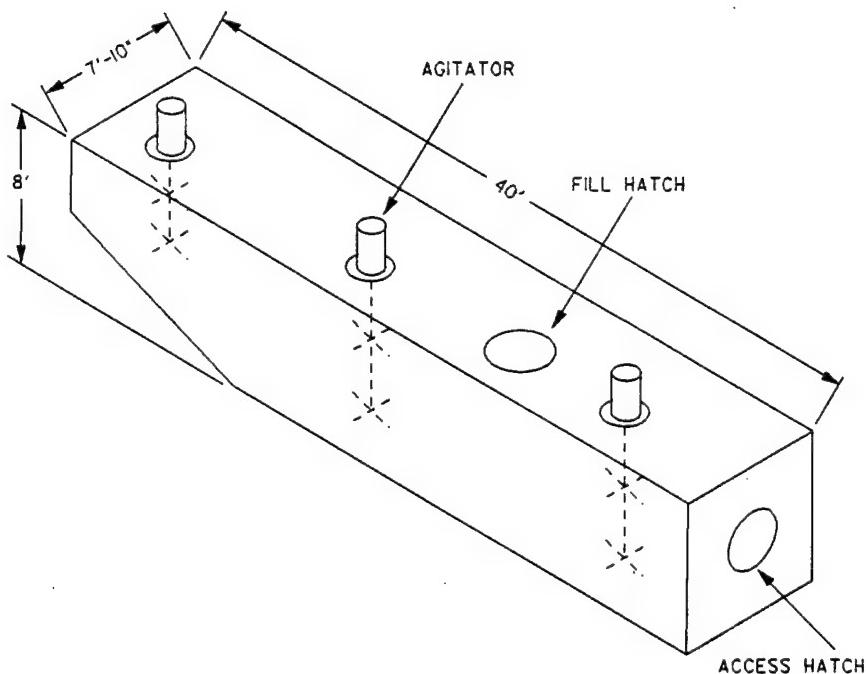
A commercial 5,000 gallon vacuum truck was leased to move all the NC fines from storage basin 9025 one mile to storage basin 3025. This truck could vacuum up the last two feet of dense NC fine slurry on the bottom of the basins. It was also used to vacuum the last NC fines from basin 3025 and put it into the reaction tanks.

### 4. Fertilizer Trucks

Two commercial fertilizer spreading trucks were used to spread the digested NC fines on the fields and pastures of Badger AAP. The 4,000 gallon trucks are



BAKER REACTION TANK



RAIN FOR RENT REACTION TANK

## FIGURE 4 REACTION TANKS

typically used to spread liquid farm manure from a farm manure storage lagoon. As the truck is driven through the field, a PTO driven pump, pumps the liquid fertilizer out of a boom over a splash plate. Typical pump rate is 100 gpm. The resultant uniform spray covers a path approximately 40 to 50 feet wide. Application rate depends on the landspeed of the truck and PTO speed. Typical landspeed was 25 mph.

## 5. Storage Basins

The nitrocellulose fines were stored over 20 years in two concrete pits of 2 million gallons capacity each. The pits or basins are divided into four quadrants each as shown in Figure 5. Nitrocellulose was evenly distributed over all quadrants. Nitrocellulose was six feet deep in the settling pit quadrants and eight feet deep in the recovered water quadrants.

## C. Methods

Specific procedures and methods were developed to process over one million pounds of NC fines. Fines were digested by potassium hydroxide hydrolysis, neutralized and the resultant liquid fertilizer landspread at Badger AAP.

### 1. NC Handling

The NC fines were pumped from the storage basins into the reaction tanks at approximately 5% solids water slurry. Excess water was decanted from reaction tanks after the slurry had settled for 4-5 hours. Decanted water was returned to the storage basin for recycle. This filling and decanting cycle was continued until the reaction tanks were filled. A sample was taken from the settled slurry to determine how much NC fines were in the reaction tank. Slurry laboratory analysis included specific gravity, percentage solids and pounds of bone-dry nitrocellulose per cubic foot of slurry. Target concentration was 18,000 pounds of bone-dry NC in a 15,000 gallon volume for a 12% solids slurry. The quantity of nitrocellulose in each batch was calculated.

### 2. NC Digestion

Potassium hydroxide (KOH), as a 50% solution, was added to the NC in the reaction tank while the slurry was agitated. The hydrolysis reaction that follows, digests the NC, converting it into a liquid fertilizer. Nitrocellulose reacts to form nitrates, nitrites and soluble cellulose. The two major chemical reactions in aqueous solution are:

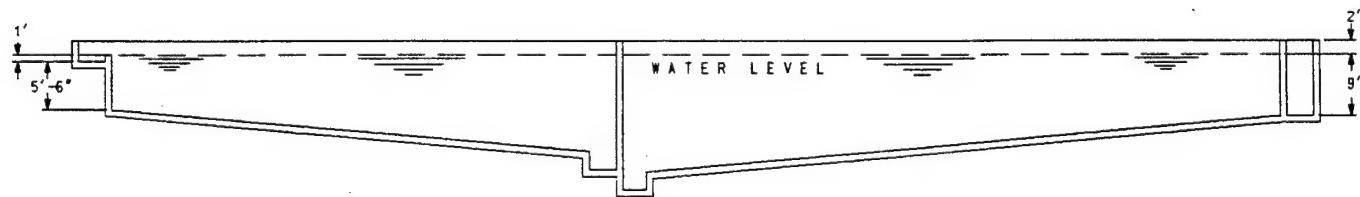
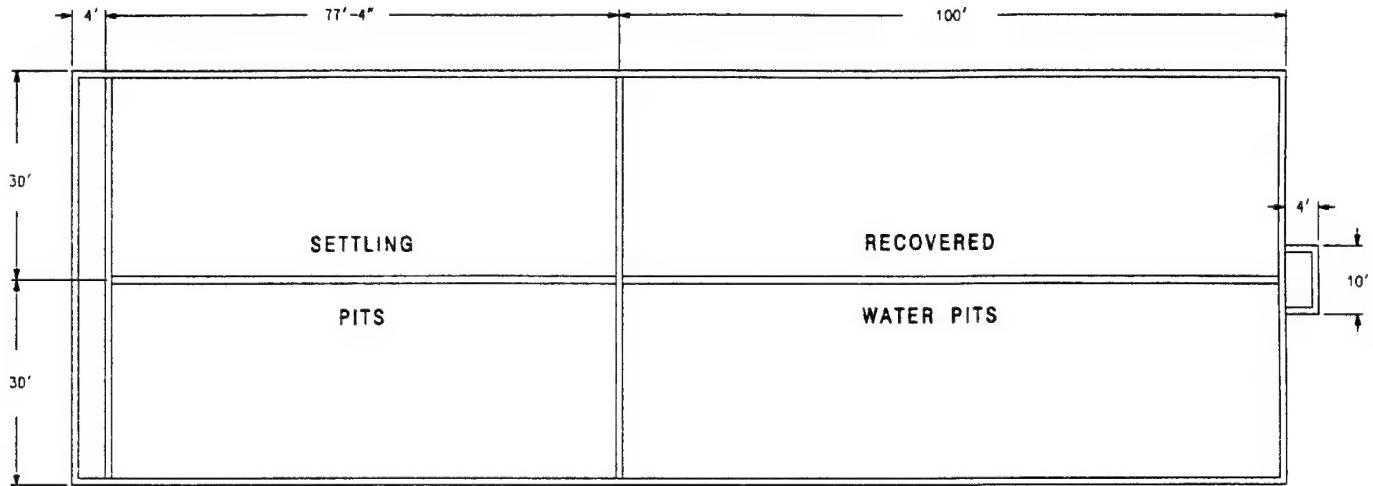


FIGURE 5    STORAGE BASINS



Six moles of KOH theoretically react with one mole of NC to form six moles of potassium nitrate or nitrite. This means 594 pounds (1 mole) of NC theoretically reacts with 336 pounds (6 moles) of KOH.

It was found by laboratory experiments that an excess of KOH must be added to drive the reactions to completion. All NC must be digested. Figure 6 depicts the effect of excess hydroxide. Seventy-five percent excess hydroxide is required to complete the digestion. Then  $336 \text{ pounds} \times 1.75 = 588 \text{ pounds}$  of KOH are needed to completely react with 594 pounds of NC. Therefore, one pound of 100% KOH was required to digest one pound of nitrocellulose. Two pounds of 50% KOH was actually added per pound of NC.

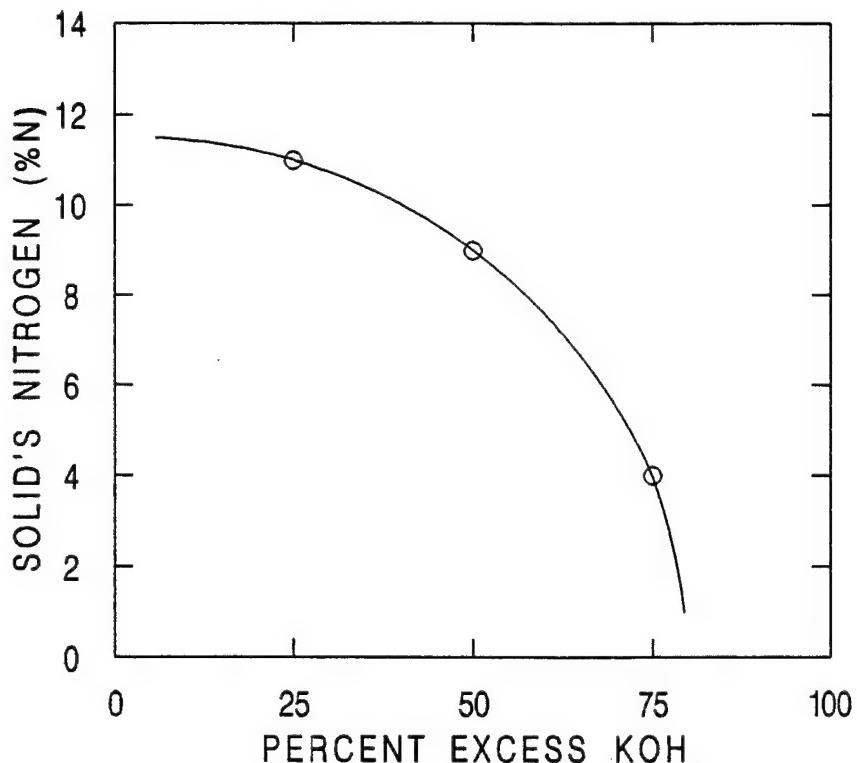


FIGURE 6  
LABORATORY DIGESTION DATA

The rate of KOH addition was controlled to maintain stable conditions within the reaction tank. Enough heat is produced by the above exothermic hydrolysis reactions to boil the reacting mass, if not controlled. KOH addition was controlled to maintain the temperature of the solution at 80°C or less. Temperature was monitored by a thermocouple in the reaction tank.

The white colored NC slurry quickly turned to a dark brown liqueur. Samples were taken from each vessel to monitor the reaction by measuring the pH and visually inspecting for unreacted NC. Unreacted NC quickly settled to form a light colored sediment in the bottom of a glass beaker. More KOH was added if any NC sediment was observed. Reactions were complete within 6 to 10 hours. At the completion of the reaction, the slurry had turned into a transparent brown liquid, without noticeable solids, of a pH over 13.5.

### **3. Digested NC Neutralization**

The reaction tank was allowed to cool, usually overnight and the pH was measured. The amount of phosphoric acid ( $H_3 PO_4$ ) required to adjust the pH to between 7 and 8 was calculated. pH adjustment is required for land application of the liquid fertilizer.  $H_3 PO_4$  was used to neutralize the liquid because phosphorus is a plant nutrient and adds to the fertilizer's food value. Final pH was recorded. pH was maintained above 7 to assure no NO<sub>x</sub> formation at the surface. The gas will dissolve readily in water and react instantly in alkali solution.

### **4. Landspreadng the Fertilizer**

As a liquid fertilizer, the digested NC consists of soluble nitrogen, potassium and phosphorus compounds in water. It is a dark brown liquid that weighs approximately 9.4 pounds per gallon. It was applied to the pastures, prairies, grasslands and fields of Badger AAP using conventional liquid fertilizer spreading trucks. Badger AAP has over 4,000 acres leased to local farmers and the U.S. Department of Agriculture, Dairy Forage Research Center (Dairy Forage). A significant amount of the liquid fertilizer was trucked to Dairy Forage's liquid manure lagoon mixed with their animal liquid manure and landspread by them at a later date.

The liquid fertilizer was pumped out of the reaction tanks to the fertilizer spreading trucks in 3,800 gallon increments. Application target rate was 500 to 1,000 gallons per acre as determined by calculating a pasture land or legume field nitrogen uptake within one growing season of 60 pounds of nitrogen per acre. The University of Wisconsin Extension recommends 40 to 160 pounds

per acre depending on the soil's potential crop yield.<sup>59</sup> Primary nutrient content was estimated at 1.2% nitrogen (N), 0.9% phosphorus (P<sub>2</sub>O<sub>5</sub>) and 8.0% potassium (K<sub>2</sub>O) or a 1:1:8 liquid fertilizer.

Records were kept of what land was spread, date of spreading, quantity of liquid fertilizer spread, area of land spread and nitrogen analysis of the liquid fertilizer.

## 5. Decontamination

The NC fines were removed from the storage basins and associated piping and sumps. All visible nitrocellulose was washed, scrubbed and scraped off the concrete and removed from the basin. Pipes were similarly cleaned of NC. After NC removal, final basin and pipe cleaning was done by high pressure water washing, followed by a mild caustic soda rinse.

A visual inspection was made of all basins and pipes to verify all visible nitrocellulose had been removed. Pipe interiors were inspected by a remote optical device. Facilities were decontaminated to a 3X (XXX) level of contamination indicating to a degree that a visual inspection of surface areas would find no contamination.

## 6. Laboratory Methods

The objective of the analysis performed by the laboratory was to ensure that the nitrocellulose was fully converted and to ensure the resultant fertilizer concentration met regulatory requirements. Analysis was performed on all batches. Samples were manifested ensuring a chain of custody from drawing the sample to reporting the results. All laboratory work was completed by Badger AAP staff chemists.

Nitrocellulose slurry samples, taken before digestion, were secured for each batch. Slurry analysis included specific gravity, percentage solids and pounds of nitrocellulose per cubic foot. Specific gravity was determined by weighing a known slurry volume. Solids content was determined by oven drying a 7 to 10 gram slurry sample. The sample was dried at 100°C. to a constant weight within 2 or 3 hours drying time. Solids content was then calculated.

Each digested batch was analyzed for nitrate and nitrite concentration, pH and reaction completion. Reaction completeness was determined by allowing the

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<sup>59</sup> K. Kelling et al (1991)

digested sample to cool to room temperature and observing any nitrocellulose solid formation on the bottom of the glass sample bottle. If any solids were observed, the reaction was not complete and required more hydroxide. Nitrate and nitrite concentration was determined using EPA Method 300.0, "Determination of Inorganic Anions by Ion Chromatography".<sup>60</sup> A Dionex DX100 ion chromatograph with computer control and output was utilized for the analysis. All nitrate and nitrite concentrations were reported as parts per million of nitrogen.

Badger AAP's laboratory determined the nitrogen level of the nitrocellulose fines. This analysis was performed using Badger's Dupont Nitrometer.<sup>61</sup> The sample is introduced into the nitrometer and decomposed by reaction with mercury and sulfuric acid. The resulting nitrogen gas is transferred to a reading tube and measured. Nitrogen values of the nitrocellulose fines ranged from 12.01 to 12.84 % N.

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<sup>60</sup> EPA (1991)

<sup>61</sup> Badger (1971)

### III. RESULTS AND DISCUSSION

The most significant result was the conversion of 1,125,000 pounds of nitrocellulose into liquid fertilizer. Conversion method was by potassium hydroxide hydrolysis of the nitrocellulose into a coffee-colored solution of nitrates and nitrites, which was then landspread as a liquid fertilizer on Badger AAP fields. Seventy-five batches of nitrocellulose or wash water were processed.

#### A Digestion Results

Sixty-eight batches of nitrocellulose were processed as described in Table 5. Quantity of nitrocellulose per batch ranged from 6,772 to 24,507 pounds. Average batch size was 16,800 pounds. The average final batch volume was  $16,500 \pm 2,000$  gallons including the 50% potassium hydroxide solution. Potassium hydroxide addition averaged  $2,600 \pm 700$  gallons. Hydroxide addition or dosage may also be expressed as a percentage of total solution weight. Dosage averaged  $18.7 \pm 3.7\%$  KOH. Nitrocellulose loading was a fairly consistent  $10.8 \pm 2.0\%$ , weight basis, solids. There were a few batches of a very light nitrocellulose solids content caused by not allowing the nitrocellulose to settle. Four batches were over 14% solid loading. Minimum loading was only 3.9% with a maximum loading of 15.1% pounds of nitrocellulose per total weight of solution.

Eight batches of process water without a measurable quantity of nitrocellulose was processed. This water was generated by recycling decant water, reaction vessel overflow, excess process water and final wash water. These batches produced a liquid fertilizer of minimal nutrient value. Process water data is shown at Table 6. Total water batches were 132,000 gallons of an average batch size of  $16,500 \pm 2,000$  gallons -- the same as the nitrocellulose batch.

Batch nitrate and nitrite concentration levels are compiled in Table 5 and Table 6. Units of measure are parts per million on a weight basis (ppmw). The tables indicate a wide range of concentration. Average nitrocellulose batch nitrite concentration was  $7,642 \pm 3,198$  ppm, ranging from a high of almost 15,000 ppm to a low of 3,200 ppm. Average nitrocellulose batch nitrate concentration was lower than the nitrite at  $6,414 \pm 2,838$  ppm. The nitrate range was somewhat less at a low concentration of 3,600 ppm to a high of 11,860 ppm. Process water concentration nitrate/nitrite concentrations were very variable ranging from none to 6,700 ppm nitrite and none to 8,520 ppm nitrate. Average data for the eight process water batches were  $1,915 \pm 2,547$  ppm nitrite and  $2,418 \pm 3,695$  ppm nitrate. All nitrate/nitrite data is reported as ppm N.

**Table 5**  
**Nitrocellulose Digestion Data**

| Batch | NC     | Volume  | KOH     | NC Vol. | Solids | Nitrite | Nitrate | pH    | Total N | NO <sub>2</sub> /NO <sub>3</sub> |
|-------|--------|---------|---------|---------|--------|---------|---------|-------|---------|----------------------------------|
|       | Pounds | Gallons | Gallons | Gallons | Wgt. % | ppmw    | ppmw    | Units | ppmw    | Ratio                            |
| 1     | 8,704  | 9,845   | 1,606   | 8,239   | 9.6    | 5,792   | 3,600   | 7.6   | 9,392   | 1.61                             |
| 2     | 13,444 | 15,994  | 2,104   | 13,890  | 9.1    | No Data | No Data | 8.9   | No Data | No Data                          |
| 3     | 10,824 | 15,944  | 1,894   | 14,050  | 7.4    | 8,410   | 5,910   | 8.2   | 14,320  | 1.42                             |
| 4     | 17,291 | 15,311  | 2,706   | 12,605  | 12.3   | 12,350  | 7,290   | 8.5   | 19,640  | 1.69                             |
| 5     | 15,070 | 15,482  | 2,358   | 13,124  | 10.6   | No Data | No Data | 8.8   | No Data | No Data                          |
| 6     | 17,422 | 15,588  | 2,727   | 12,861  | 12.1   | 11,250  | 6,970   | 8.4   | 18,220  | 1.61                             |
| 7     | 14,147 | 15,482  | 2,214   | 13,268  | 9.9    | 9,710   | 6,750   | 8.7   | 16,460  | 1.44                             |
| 8     | 16,432 | 15,311  | 2,572   | 12,739  | 11.7   | 9,180   | 6,540   | 8.8   | 15,720  | 1.40                             |
| 9     | 22,265 | 15,653  | 3,485   | 12,168  | 15.5   | 10,900  | 6,680   | 8.9   | 17,580  | 1.63                             |
| 10    | 15,000 | 18,874  | 2,347   | 16,527  | 8.6    | 9,940   | 6,840   | 7.9   | 16,780  | 1.45                             |
| 11    | 6,772  | 18,326  | 1,060   | 17,266  | 4.0    | 7,100   | 5,640   | 7.6   | 12,740  | 1.26                             |
| 12    | 16,114 | 18,044  | 2,522   | 15,522  | 9.7    | 9,350   | 6,800   | 7.8   | 16,150  | 1.38                             |
| 13    | 19,776 | 19,240  | 3,095   | 16,145  | 11.2   | 7,440   | 6,180   | 8.1   | 13,620  | 1.20                             |
| 14    | 15,186 | 17,532  | 2,377   | 15,155  | 9.4    | 9,650   | 6,570   | 7.9   | 16,220  | 1.47                             |
| 15    | 14,130 | 15,533  | 2,212   | 13,321  | 9.9    | 8,370   | 6,620   | 8.7   | 14,990  | 1.26                             |
| 16    | 15,660 | 16,336  | 2,451   | 13,885  | 10.4   | 11,200  | 7,340   | 7.9   | 18,540  | 1.53                             |
| 17    | 17,817 | 17,532  | 2,788   | 14,744  | 11.0   | 9,600   | 6,560   | 7.9   | 16,160  | 1.46                             |
| 18    | 18,999 | 15,311  | 2,973   | 12,338  | 13.5   | 10,000  | 6,560   | 7.2   | 16,560  | 1.52                             |
| 19    | 16,235 | 17,020  | 2,541   | 14,479  | 10.4   | 9,440   | 6,580   | 7.2   | 16,020  | 1.43                             |
| 20    | 13,569 | 16,336  | 2,400   | 13,936  | 9.0    | 9,230   | 6,410   | 7.9   | 15,640  | 1.44                             |
| 21    | 12,917 | 16,530  | 2,300   | 14,230  | 8.5    | 8,370   | 6,170   | 7.8   | 14,540  | 1.36                             |
| 22    | 15,095 | 17,532  | 2,362   | 15,170  | 9.4    | 6,390   | 4,330   | 8.0   | 10,720  | 1.48                             |
| 23    | 12,988 | 15,140  | 2,031   | 13,109  | 9.3    | 9,350   | 6,680   | 7.8   | 16,030  | 1.40                             |
| 24    | 15,815 | 16,165  | 2,475   | 13,690  | 10.6   | 8,160   | 4,760   | 7.4   | 12,920  | 1.71                             |
| 25    | 13,193 | 18,326  | 2,065   | 16,261  | 7.8    | 5,920   | 4,100   | 7.7   | 10,020  | 1.44                             |
| 26    | 13,765 | 15,653  | 2,154   | 13,499  | 9.6    | 8,160   | 4,760   | 8.0   | 12,920  | 1.71                             |
| 27    | 11,170 | 16,507  | 1,748   | 14,759  | 7.4    | 5,920   | 4,100   | 7.5   | 10,020  | 1.44                             |
| 28    | 16,515 | 15,995  | 2,585   | 13,410  | 11.2   | 4,840   | 3,880   | 8.0   | 8,720   | 1.25                             |
| 31    | 13,160 | 17,020  | 2,060   | 14,960  | 8.4    | 4,110   | 3,670   | 8.0   | 7,780   | 1.12                             |
| 32    | 14,955 | 18,386  | 2,341   | 16,045  | 8.8    | 6,230   | 4,450   | 8.0   | 10,680  | 1.40                             |
| 33    | 15,396 | 18,386  | 2,510   | 15,876  | 9.1    | 6,110   | 4,430   | 7.5   | 10,540  | 1.38                             |
| 34    | 19,458 | 16,336  | 3,200   | 13,136  | 12.9   | 6,140   | 4,470   | 7.7   | 10,610  | 1.37                             |
| 35    | 18,161 | 17,873  | 2,843   | 15,030  | 11.0   | 6,590   | 4,260   | 7.9   | 10,850  | 1.55                             |
| 36    | 15,760 | 15,825  | 2,467   | 13,358  | 10.8   | 5,240   | 3,950   | 7.9   | 9,190   | 1.33                             |

**Table 5 (Continued)**  
**Nitrocellulose Digestion Data**

| Batch   | NC        | Volume    | KOH     | NC Vol. | Solids | Nitrite | Nitrate | pH    | Total N | NO <sub>2</sub> /NO <sub>3</sub> |
|---------|-----------|-----------|---------|---------|--------|---------|---------|-------|---------|----------------------------------|
|         | Pounds    | Gallons   | Gallons | Gallons | Wgt. % | ppmw    | ppmw    | Units | ppmw    | Ratio                            |
| 37      | 14,607    | 14,734    | 2,286   | 12,448  | 10.8   | 4,950   | 3,980   | 7.8   | 8,930   | 1.24                             |
| 38      | 14,895    | 18,216    | 2,331   | 15,885  | 8.9    | 4,690   | 3,910   | 7.4   | 8,600   | 1.20                             |
| 39      | 19,207    | 17,019    | 3,306   | 13,713  | 12.3   | 4,510   | 4,080   | 7.8   | 8,590   | 1.11                             |
| 40      | 18,046    | 17,532    | 2,824   | 14,708  | 11.2   | 5,190   | 4,060   | 7.9   | 9,250   | 1.28                             |
| 41      | 19,392    | 18,386    | 3,035   | 15,351  | 11.5   | 5,560   | 4,150   | 8.0   | 9,710   | 1.34                             |
| 42      | 19,167    | 15,824    | 3,000   | 12,824  | 13.2   | 3,880   | 3,640   | 8.0   | 7,520   | 1.07                             |
| 43      | 18,347    | 17,926    | 2,871   | 15,055  | 11.1   | 5,030   | 4,010   | 7.7   | 9,040   | 1.25                             |
| 44      | 16,806    | 17,361    | 2,630   | 14,731  | 10.5   | 3,770   | 4,210   | 7.8   | 7,980   | 0.90                             |
| 45      | 19,530    | 16,166    | 3,057   | 13,109  | 13.1   | 6,360   | 4,120   | 7.9   | 10,480  | 1.54                             |
| 46      | 18,768    | 16,337    | 2,937   | 13,400  | 12.5   | 5,240   | 4,260   | 7.9   | 9,500   | 1.23                             |
| 47      | 16,744    | 16,166    | 2,621   | 13,545  | 11.3   | 5,090   | 4,050   | 7.8   | 9,140   | 1.26                             |
| 49      | 18,876    | 17,532    | 2,954   | 14,578  | 11.7   | 5,190   | 5,500   | 7.7   | 10,690  | 0.94                             |
| 50      | 14,221    | 17,873    | 2,226   | 15,647  | 8.6    | 4,510   | 3,840   | 7.8   | 8,350   | 1.17                             |
| 51      | 19,198    | 17,361    | 3,005   | 14,356  | 12.0   | 4,260   | 3,930   | 7.4   | 8,190   | 1.08                             |
| 52      | 21,504    | 17,873    | 3,365   | 14,508  | 13.1   | 5,080   | 4,040   | 7.6   | 9,120   | 1.26                             |
| 53      | 18,213    | 16,166    | 2,850   | 13,316  | 12.2   | 3,480   | 10,620  | 7.6   | 14,100  | 0.33                             |
| 56      | 23,025    | 16,508    | 3,604   | 12,904  | 15.2   | 4,440   | 10,800  | 7.7   | 15,240  | 0.41                             |
| 57      | 16,584    | 15,995    | 2,596   | 13,399  | 11.3   | 5,600   | 7,760   | 7.9   | 13,360  | 0.72                             |
| 58      | 19,588    | 17,361    | 3,066   | 14,295  | 12.3   | 4,440   | 10,800  | 7.8   | 15,240  | 0.41                             |
| 59      | 22,724    | 17,873    | 3,556   | 14,317  | 13.8   | 5,940   | 10,760  | 7.9   | 16,700  | 0.55                             |
| 60      | 20,252    | 17,532    | 3,170   | 14,362  | 12.6   | 13,200  | 10,740  | 7.8   | 23,940  | 1.23                             |
| 61      | 14,906    | 17,190    | 2,333   | 14,857  | 9.4    | 11,260  | 10,160  | 7.7   | 21,420  | 1.11                             |
| 62      | 21,069    | 17,361    | 3,297   | 14,064  | 13.2   | 12,340  | 10,920  | 8.0   | 23,260  | 1.13                             |
| 63      | 9,148     | 8,750     | 1,432   | 7,318   | 11.4   | 11,120  | 10,200  | 7.7   | 21,320  | 1.09                             |
| 64      | 22,223    | 16,507    | 3,478   | 13,029  | 14.6   | 12,020  | 10,920  | 7.9   | 22,940  | 1.10                             |
| 65      | 21,803    | 17,361    | 3,412   | 13,949  | 13.7   | 11,920  | 11,120  | 7.7   | 23,040  | 1.07                             |
| 66      | 20,688    | 17,500    | 4,000   | 13,500  | 12.8   | 14,960  | 11,860  | 8.8   | 26,820  | 1.26                             |
| 67      | 17,324    | 17,019    | 2,711   | 14,308  | 11.1   | 11,040  | 9,880   | 7.7   | 20,920  | 1.12                             |
| 68      | 22,530    | 17,873    | 3,526   | 14,347  | 13.7   | 12,880  | 10,780  | 7.7   | 23,660  | 1.19                             |
| 70      | 24,507    | 17,532    | 3,835   | 13,697  | 15.2   | 12,320  | 11,280  | 7.9   | 23,600  | 1.09                             |
| 71      | 20,843    | 18,044    | 3,262   | 14,782  | 12.6   | 11,260  | 10,320  | 8.0   | 21,580  | 1.09                             |
| 72      | 19,061    | 17,873    | 2,983   | 14,890  | 11.6   | 11,840  | 10,280  | 7.8   | 22,120  | 1.15                             |
| 74      | 8,050     | 7,282     | 278     | 7,004   | 12.0   | 8,200   | 8,900   | 7.6   | 17,100  | 0.92                             |
| Total   | 1,125,051 | 1,108,401 | 177,410 | 930,991 |        |         |         |       |         |                                  |
| Average | 16,305    | 16,064    | 2,534   | 13,493  | 11.1   | 7,642   | 6,414   | 7.9   | 14,056  | 1.21                             |
| St.Dev. | 4,622     | 3,411     | 820     | 2,903   | 2.1    | 3,198   | 2,838   | 0.4   | 5,608   | 0.36                             |
| Min.    | 6,772     | 7,282     | 278     | 7,004   | 4.0    | 3,480   | 3,640   | 7.2   | 7,520   | 0.33                             |
| Max.    | 24,507    | 19,240    | 4,000   | 17,266  | 15.5   | 14,960  | 11,860  | 8.9   | 26,820  | 1.71                             |

**Table 6**  
**Process Water Data**

| Batch No. | Volume  | KOH Vol | NO <sub>2</sub> as N | NO <sub>3</sub> as N | pH    |
|-----------|---------|---------|----------------------|----------------------|-------|
| Units     | Gallons | Gallons | ppmw                 | ppmw                 | Units |
| 29        | 15533   | 6       | 1310                 | 0                    | 7.4   |
| 30        | 15134   | 150     | 0                    | 0                    | 7.5   |
| 48        | 16530   | 0       | 386                  | 187                  | 8.0   |
| 54        | 17927   | 0       | 960                  | 1090                 | 7.3   |
| 55        | 18326   | 0       | 652                  | 1252                 | 7.2   |
| 69        | 18326   | 0       | 5160                 | 8180                 | 7.5   |
| 73        | 17927   | 0       | 148                  | 112                  | 7.4   |
| 75        | 12407   | 0       | 6700                 | 8520                 | 7.4   |
| Total     | 132110  | 156     |                      |                      |       |
| Ave       | 16514   | 20      | 1915                 | 2418                 | 7.5   |
| St. Dev.  | 2080    | 53      | 2547                 | 3695                 | 0.2   |
| Min       | 12407   | 0       | 0                    | 0                    | 7.2   |
| Max       | 18326   | 150     | 6700                 | 8520                 | 8.0   |

The data was analyzed in an attempt to develop some correlation to explain the nitrate/nitrite concentrations of the final fertilizer solution. Usually nitrite concentration was 20% greater than the nitrate concentration. Eight nitrocellulose and four process water batches had nitrate concentrations higher than nitrite concentration. Nitrite and nitrate concentrations were combined into a total liquid fertilizer nitrogen concentration. The ratio of nitrite to nitrate was also calculated for each nitrocellulose batch. Linear regression analysis was performed on the data of Table 5. Data was also put on scatter plots. No significant correlations could be found to explain the nitrite/nitrate concentration variability. Regression correlation coefficients were all less than 0.23. Solids loading, potassium hydroxide addition, batch volume or nitrocellulose quantity did not have a significant effect on the concentrations. The potassium hydroxide addition was a constant ratio of two pounds of 50% solution per pound of nitrocellulose. KOH was added at a constant rate to ensure the reaction temperature did not rise above 80°C. Seven nitrocellulose batches in a row (#53-#59)

produced more nitrate than nitrite. Data from these batches were analyzed to determine any significant differences with other batches. No significant differences were found.

The heavy metal content of the resultant liquid fertilizer was determined. Results are shown in Table 7 as compared to the regulatory pollutant concentrations.<sup>62</sup> Data indicates insignificant levels of heavy metal were present and well below the regulatory limit.

| Table 7<br>Heavy Metal Content<br>(units are ppm) |          |          |                  |
|---|----------|----------|------------------|
| Metal   | Batch 10 | Batch 12 | Regulatory Limit |
| Arsenic   | 0.81     | 0.82     | 41               |
| Cadmium   | 1.19     | 1.13     | 39               |
| Copper  | 3.62     | 3.57     | 1500             |
| Lead  | 2.63     | 2.97     | 300              |
| Mercury   | < 0.02   | < 0.02   | 17               |
| Nickel  | 1.17     | 1.06     | 420              |
| Selenium  | < 0.05   | < 0.05   | 100              |
| Zinc  | 17.30    | 10.80    | 2800             |

All nitrocellulose was digested. Analysis of each batch indicated no unreacted nitrocellulose being present. Potassium hydroxide amount consumed was 2,132,000 pounds of 50% solution. That is 1.895 pounds of 50% potassium hydroxide per pound of nitrocellulose actually consumed compared to the target rate of two pounds of 50% potassium hydroxide per pound of bone dry nitrocellulose. Phosphoric acid consumption, as 75% acid, was 170,000 pounds.

Negligible nitric oxide gas was emitted. Preliminary laboratory data suggested, if the phosphoric acid was added below the digested solution liquid level, only 0.5 ppm NO<sub>x</sub> would be produced. If the acid were added at the surface, data suggests 3 ppm would be produced. All phosphoric acid was added through a fill pipe at least seven feet below the liquid surface, thus negligible oxide emission. The area was continuously

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<sup>62</sup> Wisconsin Administrative Code (November 1996)

monitored with a portable gas detector.

Final fertilizer solution pH is noted in Tables 5 and 6. Initially, the final pH was held between pH 8-9. After batch #15, it was decided to lower the final pH level to pH 7-8. All later batches, except Batch #66, were within the pH 7-8 range. Batch #66 had a tank volume problem and had to be landspread at a higher pH.

#### B. Landspread Results

The digested nitrocellulose solution is a liquid fertilizer that was either landspread on Badger AAP land or emptied into Dairy Forage Research Center's liquid animal manure pit. The liquid fertilizer was mixed with the animal manure. Dairy Forage Research Center later spread the mixed manure on their fields located within Badger AAP. Total liquid fertilizer volume was 1,240,500 gallons of which 209,000 gallons (17% of total) were emptied into the Dairy Forage Research Center manure pit. One thousand seven hundred fifty-two acres were fertilized. Overall application rates were 59 pounds of nitrogen (N) per acre, 350 pounds of potassium (as K<sub>2</sub>O) per acre and 50 pounds of phosphorus (as P<sub>2</sub>O<sub>5</sub>) per acre.

A summary of landspreading data is found at Table 8 and specific data collected into Table 9. Liquid fertilizer was spread on many types of fields at Badger AAP. Most liquid fertilizer (36%) was spread on pasture land leased to local farmers for grazing beef cattle. Land was spread as the cattle grazed without segregating the sprayed area. Cattle were not disturbed. Seven hundred sixty-five acres of pasture were covered with 445,000 gallons for a coverage rate of almost 580 gallons per acre. Average nitrogen application was 92 pounds per acre and potassium application 489 pounds per acre.

**Table 8**  
**Summary Landspread Data**

| Field Type  | Volume | Nitrogen  | Potassium | Acreage               |       | Rate | Nitrogen | Potassium  |            |
|-------------|--------|-----------|-----------|-----------------------|-------|------|----------|------------|------------|
|             | %      | Gallons   | % as N    | % as K <sub>2</sub> O | Acres | %    | Gal/Acre | Pound/Acre | Pound/Acre |
| Alfalfa     | 6%     | 77,634    | 0.89      | 5.95                  | 148   | 8%   | 525      | 49         | 402        |
| Pasture     | 36%    | 444,524   | 1.75      | 9.24                  | 765   | 44%  | 581      | 92         | 489        |
| Grassland   | 3%     | 40,450    | 1.35      | 6.88                  | 70    | 4%   | 582      | 59         | 301        |
| Praire      | 4%     | 52,305    | 1.49      | 8.40                  | 125   | 7%   | 418      | 57         | 326        |
| Hay         | 30%    | 376,103   | 0.95      | 6.45                  | 548   | 31%  | 687      | 57         | 401        |
| Corn/Barley | 3%     | 40,297    | 1.12      | 6.14                  | 97    | 6%   | 415      | 46         | 235        |
| Landspread  | 83%    | 1,031,313 | 1.26      | 7.18                  | 1,752 | 100% | 589      | 59         | 350        |
| Manure Pit  | 17%    | 209,198   | 1.15      | 10.04                 |       |      |          |            |            |
| Total       | 100%   | 1,240,511 | 1.27      | 7.96                  | 1752  | 100% | 589      | 59         | 350        |

**Table 9**  
**Landscape Data**

| Batch No. | Volume Gallons | Nitrogen % as N | Potassium % as K2O | Date Spread | Field Type | Acreage Acres | Ave. Rate Gal/Acre | Nitrogen Rate Pound/Acre | Potassium Rate Pound/Acre |
|-----------|----------------|-----------------|--------------------|-------------|------------|---------------|--------------------|--------------------------|---------------------------|
|           |                |                 |                    |             |            |               |                    |                          |                           |
| 1         | 9,845          | 0.94            | 9.36               | 12 June     | Alfalfa    | 9             | 1094               | 95                       | 942                       |
| 2         | 15,994         | No Data         | 7.55               | 12 June     | Alfalfa    | 36            | 444                | 0                        | 309                       |
| 3         | 15,944         | 1.43            | 6.82               | 19 June     | Grassland  | 30            | 531                | 70                       | 333                       |
| 4         | 15,311         | 1.96            | 10.14              | 18 June     | Pasture    | 30            | 510                | 92                       | 476                       |
| 5         | 15,482         | No Data         | 8.74               | 19 June     | Manure Pit | None          | None               | None                     | None                      |
| 6         | 15,588         | 1.82            | 10.04              | 19 June     | Grass/Pit  | 12            | 520                | 87                       | 480                       |
| 7         | 15,482         | 1.65            | 8.21               | 26 June     | Manure Pit | None          | None               | None                     | None                      |
| 8         | 15,311         | 1.57            | 9.64               | 26 June     | Manure Pit | None          | None               | None                     | None                      |
| 9         | 15,653         | 1.76            | 12.78              | 30 June     | Manure Pit | None          | None               | None                     | None                      |
| 10        | 18,874         | 1.68            | 7.14               | 7 July      | Hay        | 35            | 539                | 83                       | 354                       |
| 11        | 18,326         | 1.27            | 3.32               | 10 July     | Grass/Hay  | 30            | 611                | 72                       | 187                       |
| 12        | 18,044         | 1.62            | 8.02               | 7 July      | Hay        | 30            | 601                | 89                       | 444                       |
| 13        | 19,240         | 1.36            | 9.23               | 7 July      | Praire     | 40            | 481                | 60                       | 409                       |
| 14        | 17,532         | 1.62            | 7.78               | 16 July     | Praire     | 45            | 390                | 58                       | 279                       |
| 15        | 15,533         | 1.50            | 8.17               | 16 July     | Praire     | 40            | 388                | 54                       | 292                       |
| 16        | 16,336         | 1.85            | 8.61               | 12 July     | Alfalfa    | 35            | 467                | 80                       | 370                       |
| 17        | 17,532         | 1.62            | 9.13               | 12 July     | Alfalfa    | 38            | 461                | 69                       | 387                       |
| 18        | 15,311         | 1.66            | 11.14              | 16 July     | Pasture    | 25            | 612                | 93                       | 628                       |
| 19        | 17,020         | 1.60            | 8.57               | 18 July     | Pasture    | 25            | 681                | 100                      | 537                       |
| 20        | 16,336         | 1.56            | 8.43               | 21 July     | Pasture    | 25            | 653                | 94                       | 507                       |
| 21        | 16,530         | 1.45            | 7.99               | 22 July     | Pasture    | 20            | 827                | 111                      | 607                       |
| 22        | 17,532         | 1.07            | 7.73               | 24 July     | Pasture    | 20            | 877                | 86                       | 624                       |
| 23        | 15,140         | 1.60            | 7.70               | 24 July     | Pasture    | 25            | 606                | 89                       | 429                       |
| 24        | 16,165         | 1.29            | 8.79               | 28 July     | Hay        | 25            | 647                | 77                       | 523                       |
| 25        | 18,326         | 1.00            | 6.47               | 28 July     | Hay        | 25            | 733                | 68                       | 436                       |
| 26        | 15,653         | 1.29            | 7.90               | 30 July     | Hay        | 25            | 626                | 74                       | 455                       |
| 27        | 16,507         | 1.00            | 6.08               | 30 July     | Hay        | 30            | 550                | 51                       | 308                       |
| 28        | 15,995         | 0.87            | 9.28               | 1 August    | Cut Barley | 38            | 421                | 34                       | 359                       |
| 29        | 15,533         | 0.13            | 0.02               | 30 July     | Hay        | 20            | 777                | 9                        | 2                         |
| 30        | 15,134         | 0.00            | 0.57               | 1 August    | Hay        | 20            | 757                | 0                        | 40                        |
| 31        | 17,020         | 0.78            | 6.95               | 1 August    | Cut Barley | 44            | 387                | 28                       | 247                       |
| 32        | 18,386         | 1.07            | 7.31               | 1 August    | Hay        | 30            | 613                | 60                       | 412                       |
| 33        | 18,386         | 1.05            | 7.83               | 13 August   | Hay        | 25            | 735                | 71                       | 530                       |
| 34        | 16,336         | 1.06            | 11.24              | 13 August   | Hay        | 20            | 817                | 80                       | 845                       |
| 35        | 17,873         | 1.09            | 9.13               | 15 August   | Hay        | 30            | 596                | 59                       | 500                       |
| 36        | 15,825         | 0.92            | 8.95               | 14 August   | Hay        | 25            | 633                | 54                       | 521                       |
| 37        | 14,734         | 0.89            | 8.90               | 14 August   | Hay        | 20            | 737                | 61                       | 604                       |
| 38        | 18,216         | 0.86            | 7.34               | 15 August   | Grass/Hay  | 25            | 729                | 58                       | 492                       |
| 39        | 17,019         | 0.86            | 11.15              | 20 August   | Pasture    | 25            | 681                | 54                       | 698                       |
| 40        | 17,532         | 0.93            | 9.24               | 18 August   | Pasture    | 25            | 701                | 60                       | 596                       |
| 41        | 18,386         | 0.97            | 9.47               | 22 August   | Manure Pit | None          | None               | None                     | None                      |
| 42        | 15,824         | 0.75            | 10.88              | 22 August   | Manure Pit | None          | None               | None                     | None                      |
| 43        | 17,926         | 0.90            | 9.19               | 22 August   | Manure Pit | None          | None               | None                     | None                      |
| 44        | 17,361         | 0.80            | 8.69               | 18 August   | Pasture    | 25            | 694                | 51                       | 555                       |
| 45        | 16,166         | 1.05            | 10.85              | 26 August   | Hay        | 20            | 808                | 78                       | 807                       |

**Table 9 ( Continued )**  
**Landspread Data**

| Batch No. | Volume Gallons | Nitrogen % as N | Potassium % as K2O | Date Spread  | Field Type | Acreage | Ave. Rate | Nitrogen Rate | Potassium Rate |
|-----------|----------------|-----------------|--------------------|--------------|------------|---------|-----------|---------------|----------------|
|           |                |                 |                    |              |            | Acres   | Gal/Acre  | Pound/Acre    | Pound/Acre     |
| 46        | 16,337         | 0.95            | 10.32              | 25 August    | Manure Pit | None    | None      | None          | None           |
| 47        | 16,166         | 0.91            | 9.30               | 26 August    | Manure Pit | None    | None      | None          | None           |
| 48        | 16,530         | 0.06            | 0.00               | 26 August    | Hay        | 20      | 827       | 4             | 0              |
| 49        | 17,532         | 1.07            | 9.67               | 27 August    | Manure Pit | None    | None      | None          | None           |
| 50        | 17,873         | 0.84            | 7.15               | 27 August    | Hay        | 20      | 894       | 69            | 588            |
| 51        | 17,361         | 0.82            | 9.93               | 2 September  | Hay        | 25      | 694       | 52            | 635            |
| 52        | 17,873         | 0.91            | 10.81              | 2 September  | Manure Pit | None    | None      | None          | None           |
| 53        | 16,166         | 1.41            | 10.12              | 5 September  | Pasture    | 30      | 539       | 70            | 502            |
| 54        | 17,927         | 0.21            | 0.00               | 28 August    | Hay        | 20      | 896       | 17            | 0              |
| 55        | 18,326         | 0.19            | 0.00               | 28 August    | Hay        | 20      | 916       | 16            | 0              |
| 56        | 16,508         | 1.52            | 12.53              | 5 September  | Pasture    | 30      | 550       | 77            | 634            |
| 57        | 15,995         | 1.34            | 9.31               | 5 September  | Pasture    | 30      | 533       | 66            | 457            |
| 58        | 17,361         | 1.52            | 10.14              | 9 September  | Pasture    | 30      | 579       | 81            | 540            |
| 59        | 17,873         | 1.67            | 11.42              | 9 September  | Manure Pit | None    | None      | None          | None           |
| 60        | 17,532         | 2.39            | 10.38              | 11 September | Pasture    | 35      | 501       | 110           | 478            |
| 61        | 17,190         | 2.14            | 7.79               | 12 September | Pasture    | 30      | 573       | 113           | 411            |
| 62        | 17,361         | 2.33            | 10.90              | 12 September | Pasture    | 35      | 496       | 106           | 497            |
| 63        | 8,750          | 2.13            | 9.39               | 15 September | Pasture    | 20      | 438       | 86            | 378            |
| 64        | 16,507         | 2.29            | 12.09              | 12 September | Pasture    | 30      | 550       | 116           | 612            |
| 65        | 17,361         | 2.30            | 11.28              | 13 September | Pasture    | 35      | 496       | 105           | 515            |
| 66        | 17,500         | 2.68            | 13.12              | 19 September | Pasture    | 40      | 438       | 108           | 528            |
| 67        | 17,019         | 2.09            | 9.14               | 16 September | Pasture    | 30      | 567       | 109           | 477            |
| 68        | 17,873         | 2.37            | 11.32              | 22 September | Hay        | 35      | 511       | 111           | 532            |
| 69        | 18,326         | 1.33            | 0.00               | 17 September | Pasture    | 20      | 916       | 112           | 0              |
| 70        | 17,532         | 2.36            | 12.55              | 22 September | Pasture    | 35      | 501       | 109           | 579            |
| 71        | 18,044         | 2.16            | 10.38              | 22 September | Pasture    | 35      | 516       | 102           | 492            |
| 72        | 17,873         | 2.21            | 9.58               | 22 September | Pasture    | 35      | 511       | 104           | 450            |
| 73        | 17,927         | 0.03            | 0.00               | 22 September | Alfalfa    | 30      | 598       | 1             | 0              |
| 74        | 7,282          | 1.71            | 2.19               | 25 September | Cut Corn   | 15      | 485       | 76            | 98             |
| 75        | 12,407         | 1.52            | 0.00               | 22 September | Pasture    | 20      | 620       | 87            | 0              |
| Total     | 1,240,511      |                 |                    |              |            | 1752    |           |               |                |
| Average   | 16,111         | 1.27            | 7.96               |              |            | 23      | 508       | 59            | 350            |
| St.Dev.   | 3,293          | 0.66            | 3.55               |              |            | 13      | 276       | 39            | 254            |
| Min.      | 7282           | 0.00            | 0.00               |              |            | 9       | 387       | 1             | 0              |
| Max.      | 19,240         | 2.68            | 13.12              |              |            | 45      | 1094      | 116           | 942            |

Five hundred and fifty acres of hay land was covered with 376,000 gallons of liquid fertilizer. This was 30% of total volume landspread. These hay fields are sold to local farmers who cut and remove the hay crop. Liquid fertilizer was spread shortly after the hay was cut. This enabled a second crop to receive the benefit of the fertilizer. Fertilizer application rate averaged 687 gallons per acre resulting in 57 pounds of nitrogen per acre application and 401 pounds of potassium per acre. Second crop application areas were observed to have a thicker, more dense and darker green growth than the unapplied hay land.

Fertilizer was applied to 70 acres of Badger AAP grassland or 4% of the landspreading area. Grassland is open area currently not leased as pasture or hay fields, but so leased in the past or potentially leasable in the future. Some of this land includes perimeter fence grassland mowed by maintenance personnel maintaining plant security. This land including roadside grassland acreage (15 August) was landspread with 40,000 gallons at a rate of 582 gallons per acre. Nitrogen application was 59 pounds per acre and potassium application was 301 pounds per acre.

Several Dairy Forage Research Center's alfalfa fields were spread within a few days of their first cutting. Second and third alfalfa cuttings from the areas of fertilizer application produced significantly more forage tonnage than the areas not applied. Improved tonnage was observed by the increased plant density and plant height of the applied areas. Seventy-eight thousand gallons were spread on 148 acres of alfalfa for an application rate of 525 gallons per acre. Nitrogen application averaged 49 pounds per acre and potassium application was 402 pounds per acre.

One hundred and twenty-five acres of prairie restoration were spread with 52,000 gallons of liquid fertilizer. This prairie landspread on 7 and 15, July was easily distinguished from adjacent prairie not fertilized. The lush, dark green grasses were obvious. Application rate averaged 418 gallons per acre for a potassium application rate of 326 pounds per acre and a nitrogen application rate of 57 pounds per acre.

Two barley fields and one cornfield were spread after the crop had been cut off. The barley fields were subsequently seeded with alfalfa. The three fields totaled 100 acres, spread with 40,000 gallons, for an average potassium application of 235 pounds per acre and a nitrogen application rate of 46 pounds per acre.

Specific disposal for each digestion batch is found in Table 9. Volume, date spread, acreage, type field spread, spreading rate, potassium/nitrogen application rate and potassium/nitrogen nutrient loading are listed for each batch. An average batch of 16,200 gallons was spread on a 23 acre field at a spreading rate of 589 gallons per acre, at a nitrogen application rate of 59 pounds per acre and potassium application rate of 350 pounds per acre. Standard deviation, minimum and maximum data for each parameter, is also noted in the table.

Liquid fertilizer phosphorus nutrient level was not analyzed. Calculations indicate a nutrient level of 0.94% phosphorus (as P<sub>2</sub>O<sub>5</sub>) resulting in an application rate of approximately 50 pounds of phosphorus per acre.

C. **Regulatory Discussion**

The principle regulatory document for disposal of the nitrocellulose fines is the Environmental Protection Agency's rules as published in 40 CFR 261.2(e)(1)(ii) and (f).<sup>63</sup> Rules specify materials are not solid wastes when they can be shown to be recycled by being used or reused as effective substitutes for commercial products. Record keeping guidelines have been established for facilities to support their claims of recycling materials. Documentation has been developed supporting that the nitrocellulose fines were not hazardous waste, because they were being recycled and the process for converting the nitrocellulose fines into a fertilizer was recycling and not treatment. Guidelines consist of the four points noted in the following paragraphs.

1. **Known Market**

A known market for the materials were demonstrated. The liquid fertilizer generated from digestion of nitrocellulose was landspread on Badger AAP land leased or potentially leasable. Each parcel of leased or leasable land has a tract management plan stipulating the required soil treatments over the leased period. Plans indicate the maintenance fertilizers to be used. If nitrocellulose liquid fertilizer was not available, other commercial fertilizer would have been purchased for tract application.

2. **Equipment**

Badger AAP had the necessary equipment for conducting the recycling as documented in this report. A knowledgeable company was contracted to perform the disposal. The proper quality and quantity of reaction tankage, pumps and trucks were leased to successfully complete the operation.

3. **Effective Substitute**

This report documents that the generated liquid fertilizer was an effective substitute for commercial fertilizer. Data indicates the actual application rates for the various fields landspread. Generally accepted nutrient application rates are shown in the following Table 10.<sup>64</sup> These application rates were developed by the Soils and Plant Analysis Laboratory, University of Wisconsin - Extension

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<sup>63</sup> 40CFR 261 (July 1995)

<sup>64</sup> K. Kelling, et al (1991)

and are known to the Wisconsin farming public as the University of Wisconsin Soil Test Recommendations. The range of recommended optimum application is keyed to the crop yield goal. More nutrient is required if a higher crop yield is desired. Comparison to the actual application rates are also shown in Table 10.

**Table 10**  
**Annual Fertilizer Application**  
 (units are lbs/acre)

| Field Type  | Nitrogen as N |        | Potassium as K <sub>2</sub> O |        | Phosphorus as P <sub>2</sub> O <sub>5</sub> |        |
|-------------|---------------|--------|-------------------------------|--------|---|--------|
|             | Recommended   | Actual | Recommended                   | Actual | Recommended                                 | Actual |
| Alfalfa     | 30            | 49     | 100-350                       | 402    | 25-90                                       | 45     |
| Pasture     | 40-160        | 92     | 60-240                        | 489    | 25-60                                       | 50     |
| Grassland   | 40-160        | 59     | 60-240                        | 301    | 25-60                                       | 50     |
| Prairie     | 80-160        | 57     | 60-240                        | 326    | 25-60                                       | 36     |
| Hay         | 40-160        | 57     | 60-240                        | 401    | 25-60                                       | 59     |
| Corn/Barley | 80-200        | 46     | 20-60                         | 235    | 30-120                                      | 51     |
| Overall     |               | 59     |                               | 350    |   | 50     |

Table 8 indicates the nitrocellulose liquid fertilizer is a very effective substitute for commercial fertilizer, applying a significant amount of or more than the recommended annual fertilizer application. The excess potassium is not an environmental hazard.<sup>65</sup> It is tightly held by soil particles and has little potential of leaching into ground or surface waters.

#### 4. Records

Documentation in this report indicates how, where, and in what volumes the nitrocellulose liquid fertilizer was applied.

The EPA has established the above guidelines to confirm a legitimate recycling activity. It is shown the nitrocellulose liquid fertilizer is not an ineffective or marginally effective fertilizer. Excessive amounts were not used and the quantities used effectively replaced commercial fertilizer. Application could have been on lands outside Badger AAP, however with sufficient land available within Badger AAP, the Army opted to only spread within Badger AAP. Project documentation is complete.

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<sup>65</sup> Dennis Keeney, et al (1975)

The nitrocellulose digestion operation also has the potential to release hazardous nitric oxide gas. Preliminary laboratory data suggested adding materials below the processing liquid level will eliminate gas generation. Calculations predicted only three pounds of NO<sub>x</sub> would be emitted during the entire operation. Wisconsin Department of Natural Resources were asked of the need for an air discharge permit and advised that a permit would not be required.

#### D. Economics

The subcontracted cost of this project was \$837,500 to digest 1,125,000 pounds of nitrocellulose into a liquid fertilizer. Cost can be detailed as shown in Table 11. Administrative cost included the contractor's work plan preparation, health and safety efforts, analytical work and profit. Labor consisted of two operators and an on-site project manager. Minimal additional labor was necessary to vacuum nitrocellulose from the storage basins.

Other project costs were incurred by Badger's Operating Contractor - Olin Corporation. These costs included maintenance labor to remove the storage basin covers to allow the subcontractor access to the basin, electric work to energize reaction vessel agitators and other minor tasks. Significant analytical work was performed by Badger AAP's laboratory. Project engineering, supervision, inspection and overhead costs were also expended. Table 11 details the cost items. These demonstration project in-house costs would not be expected to be as high for future projects.

| Table 11<br>Project Cost Data |              |          |
|-------------------------------|--------------|----------|
| Element                       | Cost         | Cost/Lb. |
| Chemicals                     | \$ 484,000   | \$ 0.43  |
| Equipment Rental              | 150,000      | 0.13     |
| Labor/Overhead                | 136,000      | 0.12     |
| Travel/Per Diem               | 30,000       | 0.03     |
| Administration                | 30,500       | 0.03     |
| Subcontract Total             | \$ 837,500   | \$ 0.74  |
| In-house Labor                | 82,000       | 0.07     |
| Other In-house Costs          | 133,000      | 0.12     |
| In-house Total                | \$ 215,000   | \$ 0.19  |
| Total                         | \$ 1,052,500 | \$ 0.93  |

E. Time Frame

The conversion contractor arrived on-site May 12, 1997 to begin setting up the equipment. First chemical delivery was June 5. Conversion operations began the next day with the first batch being loaded into a reaction tank. This first batch was landspread on June 12. Operations continued Monday through Friday with occasional Saturday and Sunday work. Fourth of July and Labor Day were taken off. The last batch was landspread September 25. Final inspection was held September 30 after cleanup was complete. Demobilization was complete October 2, 1997. The project was completed in 110 working days of which 83 days included digestion or landspreading operations.

## IV. SUMMARY AND CONCLUSIONS

This engineering report documents the conversion of 1,125,000 pounds of bone-dry nitrocellulose fines that had been stored for over twenty years at Badger AAP. Conversion method was potassium hydroxide hydrolysis to digest the nitrocellulose, followed by phosphoric acid neutralization. The resultant liquid fertilizer (1,240,000 gallons) was landspread on Badger AAP fields. Project conclusions are:

- It was demonstrated alkaline hydrolysis, nitrocellulose conversion with neutralization and landspreading as a liquid fertilizer is a proven high volume, economical conversion method.
- Excess potassium hydroxide is required to completely digest the nitrocellulose.
- Phosphoric acid neutralization of the hydrolysis products resulted in a liquid fertilizer of the following plant nutrient value - 1.3% nitrogen (N), 8.0% potassium (K<sub>2</sub>O) and 0.9% phosphorus (P<sub>2</sub>O<sub>5</sub>).
- The conversion method met all applicable federal and state safety and environmental regulations.

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